

# SAMPLING AND ANALYSIS OF PERCHLORATE IN FERTILIZER (Revision 3)

Prepared for

### Perchlorate Study Group

Shreveport, Louisiana

Prepared by

**TRC** 

Irvine, California

#### Participating Laboratories

American Pacific Corporation - Utah
Del Mar Analytical - California
Montgomery Watson Laboratories - California
Thiokol Corporation - Utah
Air Force Research Laboratory - Wright Patterson AFB, Ohio
United States EPA National Exposure Research Laboratory
Ecosystems Research Division - Georgia
United Technologies Corporation - California



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### **TRC**

Irvine, California

Project No. 98-346 July 1999

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#### 1.0 INTRODUCTION AND BACKGROUND

- 1. The presence of naturally-occurring perchlorate has been identified in sodium- and potassium-nitrate mined from caliche deposits in Chile (i.e., in Chilean saltpeter). Chilean saltpeter is used as a nitrate fertilizer throughout the world. Schilt<sup>(1)</sup> describes observations of perchlorate in Chilean saltpeter as far back as 1896, with concentrations ranging from trace amounts to just under 7 percent. Levels of perchlorate of 1 to 1.5 percent in refined and crude Chilean saltpeter, respectively, measured in 1914 were also described (Schilt, 1979). A study in 1972 attributed poor soybean growth to elevated concentrations of perchlorate in Chilean nitrate fertilizer. The relatively broad range of perchlorate in Chilean saltpeter concentrations was attributed to varying degrees of the liquid recycling during the production of the nitrate salts, and the possible nonuniform distribution of naturally-occurring perchlorate in the caliche. Review of available literature and recent limited testing of selected samples of chemical fertilizer indicate the presence of perchlorate.
- 2. Based on the above-referenced literature, data and the results of the limited fertilizer sampling and analysis of California fertilizers for perchlorate conducted in August of 1998, additional studies were performed to determine if perchlorate is typically present in nitrate type fertilizers. In order to confirm the above results and also to obtain more information on the presence of perchlorate associated with the use of fertilizers, the following activities were completed, and are presented in this report:
  - Confirmatory sampling and analysis of previously tested products.
  - Collection and analysis of fertilizers nationwide.
  - Collection and analysis of fertilizer raw materials.

#### 2.0 SAMPLING AND ANALYSIS

#### 2.1 SAMPLE COLLECTION

1. To confirm the results of the recent limited perchlorate analysis results indicated above, samples were obtained from different production lots of previously tested California fertilizers. These samples were purchased from various commercial suppliers and retail sources.

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<sup>(1)</sup> Schilt, Alfred, "Perchlorate Acid and Perchlorates, Fredrick Smith Chemical Company," 1979.

- 2. To obtain information on the potential presence of perchlorate associated with the use of chemical fertilizers, additional samples were collected from the Kansas City, Missouri and Long Island, New York areas. Samples of the fertilizers shown in Table 1 were collected from various commercial suppliers and retail sources.
- 3. Also, as part of this study, samples of two raw materials used in chemical fertilizer production were also collected. Caliche (Chilean nitrate) was obtained from the Aconcagua Nitrate Mine in Calama, Chile by TRC. A langbeinite (a potassium/magnesium sulfate mineral) ore sample (crushed) was provided by IMC Minerals in Albuquerque, New Mexico.
- 4. The fertilizers and raw material samples were collected under chain-of-custody procedures and forwarded to the primary laboratory for analysis.
- 5. The primary laboratory prepared extracts of each sample, which were then shipped to six outside laboratories for confirmatory analysis.

#### 2.2 ANALYSIS

#### 2.2.1 ION-CHROMATOGRAPHY

- 1. The primary laboratory (Del Mar Analytical, Irvine, California) analyzed the fertilizers using the California Department of Health Services (DHS) Method of Ion-Chromatography for Perchlorates (see Attachment 1). Table 1 provides a summary of the Ion-Chromatography perchlorate results.<sup>(2)</sup>
- 2. As discussed above, six outside laboratories analyzed the perchlorate extracts for confirmation purposes. However, these laboratories did not all perform the analyses using the California DHS Method for perchlorates. Table 2 provides a list of the methods used by each laboratory and the differences between the method used and the California DHS Method.
- 3. Table 1 provides a summary of the results of the analyses conducted by the outside laboratories discussed above. As noted in Table 1, the outside laboratories analyzed a 1:10 extract. The results in Table 1 for the outside laboratories were adjusted by a factor of ten to allow comparison with the primary laboratory data. Copies of the laboratory reports are provided in Appendix A.

<sup>(2)</sup> Perchlorate sample analyses results from the United States Environmental Protection Agency were not yet available at the time of publishing this report. The report will be updated when USEPA results become available.

#### 2.2.2 TRIPHENYLSTIBONIUM TITRATION

1. As part of this study, one set of the fertilizer extracts and various control samples prepared by TRC were analyzed using the triphenylstibonium titration method after purification of the extracts using ion exchange resin adsorption. The purpose of performing the titration study was to chemically confirm the presence of perchlorate in the extracts. The triphenylstibonium titration method has been shown to be highly specific for perchlorate, and therefore can be used to confirm the Ion-Chromatography results, which are highly accurate, but may be less specific in detecting perchlorate. Table 1 also includes a summary of the triphenylstibonium titration results.

#### 3.0 DISCUSSION

- 1. The results of the sampling and analysis of the fertilizers indicated that perchlorate was present at varying levels in approximately 27 out of the 28 fertilizers tested (i.e., 96 percent). Please note that four fertilizer samples were analyzed in duplicate, thus there are 32 entries on Table 1. Raw materials samples, caliche and langbeinite ore, tested positive for perchlorate. The detectable perchlorate levels in these fertilizers ranged from approximately 2 parts per million (ppm) to over 11,000 ppm with most being greater than 500 ppm. The perchlorate concentration averaged roughly one-half of one percent (0.5 percent or 5,000 ppm), as measured in the 27 fertilizers that were positive for perchlorate. These results indicate that perchlorate levels can vary significantly between fertilizer brands and formulation types.
- 2. The results of the analysis of different production lots of the previously tested fertilizers showed significant variations between the different lots, indicating either variations in production practices or perchlorate levels in the raw materials. Both raw materials tested, caliche and langbeinite, contained elevated levels of perchlorate; 29,000 ppm and 15,000 ppm (i.e., 2.9 and 1.5 percent), respectively.
- 3. A comparison of the perchlorate results from the primary laboratory and the outside laboratories shows an excellent correlation. As indicated in Table 1, the standard deviations are very low, indicating a high level of precision. Table 1 also indicates the various duplicate samples and their results. The duplicate samples were found to generally agree within normal laboratory limits (i.e., ±50 percent), with the exception of one duplicate sample PS-17/PS-24 which showed an unexplained significant difference. This difference is most likely due to an error in the extract preparation, since all of the laboratories had similar results for these samples.

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4. The results of the triphenylstibonium titration appear to generally agree with the ion-chromatography results, although the method appears to slightly over estimate the perchlorate levels. Quality control samples submitted to the laboratory containing known levels of perchlorate, chlorate, chlorite, ammonium nitrate and thiosulfate showed no interference with the perchlorate analysis. Additional duplicate samples submitted also showed good correlation, within the expected limits.

#### TABLE 1

### FERTILIZER PERCHLORATE LIQUID CHROMATOGRAPHY TESTING PRELIMINARY RESULTS

	MANUFACTURER	BRAND NAME	N-P-K <sup>(I)</sup> RATIO	PRIMARY CONSTITUENT	TRIPHENYL- STIBONIUM TITRATION	ION- CHROMATO- GRAPHY DEL MAR		SPLIT SAM	PLE ANALYSIS	RESULTS (1	mg/kg) <sup>tti</sup>			ISTICAL LUATION	PERCENTAGE OF PERCHLORATE (%)
			KAIIO	}	(mg/kg) <sup>(1)</sup>	ANALYTICAL RESULTS (mg/kg)	LAB I	LAB 2	LAB 3	LAB 4	USAF	USEPA <sup>(14)</sup>	MEAN®	STANDARD DEVIATION (13)	,
Obtained	1/27/99 Kansas City. N	Aissouri		<u> </u>											
PS-11	Scott's	Miracle-Gro Lawn Food	36-6-6	Potash derived from Potassium Nitrate	8,600	7,4(X)	8,160	6,260	8,800	8,290	7,068		7,663	930	0.77
PS-13	Shulte	Rose Pius	19-24-24	Potassium Nitrate	<50	13	<6.0	<40	<0.8	9,6	611		9.2	8	0.0009
PS-10	Acme	Stump Remover	NA	Potassium Nitrate	<50	< 0.4	<6.0	<40	<0.4	<3.0	an		4.98	8	<.0003
	Sudbury	Potash	0-0-44	Potassium Chloride	5,440	4,800	4,730	8.830	5,000	4,920	4,369		5,442	1,674	0.54
PS-05	Peters	All Purpose Plant Food	20-20-20	Potassium Nitrate	7,150	6,3(X)	6,7(X)	4,760	6,200	6.360	6,898		6,203	754	0.62
PS-14	Jobe's	Plant Food Spikes	16-2-6	Potassium Nitrate	10,350	8,100	9,480	8,640	9,(KX)	9,920	6,896		8,673	1,077	0.87
PS-01	Fertilome	Start-N-Grow Plant Food	18-6-12	Potassium Nitrate	5,9(X)	4,7(8)	4,680	5,150	5,3(X)	4,710	4,872		4,902	264	0.49
PS-08	Osmocote	Vegetable and Bedding	14-14-14	Ammonium Nitrate	1,250	940	1,090	968	970	1,010	803		964	94	0.09
PS-12 <sup>(6)</sup>	Osmocote	Plant Food Vegetable and Bedding	14-14-14	Ammonium Nitrate	750	540	636	597	530	580	526		568	44	90.0
PS-()2		Plant Food Supreme Gardens	7/7/07	Nitrate of Soda	2,950	3,200	3,490	2.360	3,1(X)	3,240	3.077		3.078	381	0.31
PS-09 <sup>(5)</sup>		Supreme Gardens	7/7/07	Nitrate of Soda	5,200	3,9(x)	4,380	5,120	4,3(X)	4,380	4,049		4,355	422	0.44
PS-03	Peters	Lawn Food	38-4-4	Potassium Nitrate	9,9(x)	8,6(X)	8,950	7,730	9,4(0)	8,860	7,678		8,536	l.	0.85
PS-()4	HiYield	Nitrate of Soda	16-0-0	Nitrate of Soda	6,800	7,4(x)	7,620	6,300/LL840 <sup>cto</sup>	8,000	7,660	6,908		7.637	761	
Obtained	1/28/99 Long Island,	New York													
PS-15	Ringer	Lawn Restorer	10/2/06	Nitrate of Soda	6,140	5,300	6,060	5,190	6,100	6,080	4,673		5,567	663	0.56
PS-21 <sup>(8)</sup>	Ringer	Lawn Restorer	10/2/06	Nitrate of Soda	3,4(x)	2,4(%)	2,960	2,560	2,9(X)	3,000	2,376	121, 131	2,695	28	0.27
PS-20	Frank's	Grow	15-30-15	Muriate of Potash	6,250	5,4(X)	5,380	10,0XX)	4,800	5,560	7,098		6,37	1,930	0.62
PS-16	Peters	All Purpose Plant Food	20-20-20	Potassium Nitrate	6,980	7,3()	7,510	5,640	7,4(X)	7,210	6,194		6,876	776	0.7
PS-19	Osmovote	Vegetable and Bedding Plant Food	14-14-14	Ammonium Nitrate	3,100	2,500	2,650	3,270	2,7(X)	2,660	2,053		2,639	39	0.26
PS-18	Osmocote	Outdoor and Indoor Plant	18-6-12	Ammonium Nitrate	740	6(X	733	832/993 <sup>(10)</sup>	750	690	904		765	25	80.0
PS-23	Vigoro	Tomato and Vegetable Plant Food	10/8/14	Muriate of Potash, Sul Po Mag	450	360	464	738	340	430	514		47-	14	0.05
PS-17	Jonathan Green	Fall Fertilizer	10/18/20	Muriate of Potash	<50	< 0.4	<6.0	<40	2.2	<3.0	412		5	3	0.0005
PS-24 <sup>171</sup>	Jonathan Green	Fall Fertilizer	10/18/20	Muriate of Potash	2,850	2,4()(	2,590	2,840	2,2(X)	2,480	2,530		2,50	7 21:	0.25
PS-22	Scotts	Miracle-Gro Lawn	31-3-9	Muriate of Potash	1,700	1,300	1,549	2,646	1,400	1,530	1,995		1,73	7 50	0.17
	from California Source	Fertilizer :e		·		***************************************	<u></u>		<u> </u>	<u> </u>		<del></del>		<u> </u>	·
PS-31	CNC <sup>(3)</sup>	Champion Potassium	13.5-0-45		16,800	12,000 (7,500)	13,290	14,380	13,000	15,100	11,749		10,72	5,94	7 1.07
PS-33	Best <sup>(3)</sup>	Nitrate K-Power Potassium Nitrate	13.75-()-46		8,250	7,100 (27,500)	7,563	2,600	7,400	7,870	7,201		8,32	2 4,45	0.83
	Grow More Corp <sup>(2)</sup>	Grow More	6/30/30			2,5(X) (<4,(XX))(4	2,599	3,623	2,2(X)	2,490	3,135		2,21	2,10	0.22
PS-25,	Best <sup>(1)</sup>	Triple Sixteen	16-16-16			4,200 (18,000)	4,223	3,680	4,000	4,310	3,727		5,17.		* **
PS-26	Bandini <sup>(3)</sup>	Sui Po Mag	()-()-22			3,(XX),(15,(XX),E	3,073	4,810	3,2(X)		2,834		4,34	·	
PS-27	Plant Marvel	Natriculture	12/31/14	Potassium Nitrate	6,3tX			7,120	5,5(X)		6,246		6,21		
PS-29	Dexol	Stump Remover	Unknown	Unknown	5,800	<del></del>	5.189	5,290	5,4(X)		4,576		5.12		· · i
PS-32	K Power	Mini Prilis	13.75-0-46	Chileans Nitrate	8,2(X	<del> </del>	6,381	6,300	6,100	6,560	5,862		6,25	·	0.63
PS-06	Peters	All Purpose	20-20-20	Potassium Nitrate	3,400	·	<del></del>	3,680	3,(XX)				3,21		
<del></del>		Control Sample 2/8/99			1	1	<u> </u>	<u> </u>		<del></del>	<u> </u>		<u> </u>	1	L
PS-34		Callche (Chilean Nitrate	Unknown	Sodium Nitrate	32,800	30,08	26,310	32,980	27,133	30,900	26,120	,	28.88	5 2.82	5 2.89
PS-30	IMC Group	Source) Langbeinite Ore	Unknown	Magnesium, Sulfur,	15,600		14,3(X)	20,090	14,0XX				15,24		
LCS-I**		**	Unknown	Potassium	90		96	99	10X				9		0.009
LCS-2(9)		NA	Unknown		105		104	94	99		98		9		0,009
Blank			Unknown		<51		<(),(X)3	<0,003	<().()()4	<0.003			0.00		

Laboratory extract results (mg/L) were converted to mg/kg units for comparative purposes, by multiplying the result by 10 to account for the 1:10 dilution used to prepare the extracts.

<sup>12)</sup> Nitrogen, Phosphorus and Potassium Levels.

<sup>(3)</sup> Repeat of Prior Testing.

<sup>(4)</sup> Prior Testing Results.

<sup>(5)</sup> Duplicate for PS-02,

m Duplicate for PS-08.

Ouplicate for PS-17.

on Duplicate for PS-15.

<sup>&</sup>lt;sup>420</sup> Laboratory Control Standard of 100 ppb Quality Control Samples. <sup>(98)</sup>

<sup>(18)</sup> Interim duplicate sample analysis.

<sup>(</sup>ii) Due to interferences, no value was reported.

Mean values calculated from results obtained by Del Mar Analytical, Labs 1 through 4 as noted, and the United States Air Force. For samples with duplicate analyses, the duplicate results were averaged before calculating the overall mean and standard deviation.

<sup>513)</sup> Standard deviation values calculated from results obtained by Del Mar Analytical, Labs 1 through 4 as noted, and the United States Air Force.

For samples with duplicate analyses, the duplicate results were averaged before calculating the overall mean and standard deviation.

(14) Perchlorate sample analyses results from the United States Environmental Protection Agency will be provided after completion of Quality Assurance/Quality Control (QA/QC) evaluation by the

# TABLE 2 SUMMARY OF ION-CHROMATOGRAPHY METHODS

LABORATORIES	SIGNIFICANT DIFFERENCES BETWEEN OUTSIDE LABORATORY PROCEDURES AND DHS PERCHLORATE METHOD
Del Mar Analytical	Used 4 mm Standard Bore Column (AS-11)
American Pacific Corporation (Lab 1)	Used 4 mm Standard Bore Column (AS-16)
United Technologies Corporation (Lab 2)	<ul> <li>Used 4 mm Standard Bore Column (AS-11)</li> <li>Used 100 mM NAOH without Ion-Suppressor</li> </ul>
Montgomery Watson Laboratories (Lab 3)	Used 4 mm Standard Bore Column (AS-11)
Thiokol Corporation (Lab 4)	Used 4 mm Standard Bore Column (AS-11) Used 100 mM NAOH with Ion-Suppressor AS-RS-ULTRA
U.S. Air Force Research Laboratory	Used 4 mm Standard Bore Column (AS-16)
U.S. EPA	To be provided after completion of Quality Assurance/Quality Control Evaluation

#### APPENDIX A

#### LABORATORY REPORTS

- **A.**1 DEL MAR ANALYTICAL
- A.2 AMERICAN PACIFIC CORPORATION
- UNITED TECHNOLOGIES CORPORATION MONTGOMERY WATSON LABORATORIES A.3
- A.4
- A.5 THIOKOL CORPORATION
- UNITED STATES AIR FORCE ARMSTRONG LABORATORY A.6
- UNITED STATES ENVIRONMENTAL PROTECTION AGENCY(3) A.7
- CHEMICAL TESTING SERVICE (PERCHLORATE TITRATION RESULTS) A.8

<sup>(3)</sup> Perchlorate sample analyses results from the United States Environmental Protection Agency will be provided after completion of Quality Assurance/Quality Control (QA/QC) evaluation.

APPENDIX A.1
DEL MAR ANALYTICAL



2852 Alton Ave., Irvine, CA 92606 1014 E. Cooley Dr., Suite A. Colton, CA 92324 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 9484 Chesapeake Dr., Suite 805, San Diego, CA 92123 9830 South 51st St., Suite B-120, Phoenix, AZ 85044 (949) 261-1022 FAX (949) 261-1228 (909) 370-4667 FAX (909) 370-1046 (818) 779-1844 FAX (818) 779-1842 (619) 505-9596 FAX (619) 505-9688 (602) 785-0043 FAX (602) 785-0851

#### LABORATORY REPORT

Prepared For:

TRC Environmental Solutions

21 Technology Drive Irvine, CA 92618

Attention:

Richard Scott

Project: 98-346

Lockheed

Sampled:

2/24/99

Received:

2/25/99

Reported:

3/9/99

This laboratory report is confidential and is intended for the sole use of Del Mar Analytical and its client. This entire report was reviewed and approved for release.

CA ELAP Certificate #1197 AZ DHS Licence #AZ0428

**DEL MAR ANALYTICAL** 

Fred Haley Project Manager

Results pertain only to samples tested in the laboratory. This report shall not be reproduced, except in full, without written permission from Del Mar Analytical.

IB03512.TRC <1 of 6>



# Del Mar Analytical

2852 Alton Ave. Irvine. CA 92606 1014 E. Cooley Dr., Suite A. Colton, CA 92324 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 9484 Chesapeake Dr., Suite 805, San Diego, CA 92123 9830 South 51st St., Suite B-120, Phoenix, AZ 85044 (949) 261-1022 FAX (949) 261-1226 (909) 370-4667 FAX (909) 370-1046 (818) 779-1844 FAX (818) 779-1843 (619) 505-9596 FAX (619) 505-9688 (602) 785-0043 FAX (602) 785-0851

TRC Environmental Solutions

21 Technology Drive

Irvine, CA 92618 Attention: Richard Scott Client Project ID: 98-346

Lockheed

Sample Descript: Solid First Sample #: IB03512 Sampled:

Feb 24, 1999

Received: Extracted:

Feb 25, 1999 Mar 6-8, 1999

Analyzed: Reported:

Mar 6-8, 1999 Mar 9, 1999

#### PERCHLORATE (EPA 300.0 Mod.)

Laboratory Number	Sample Description	Reporting Limit mg/Kg (ppm)	Sample Result mg/Kg (ppm)	QC Batch
1B03512	PS-01	400	4,700	IC06D31S
IB03513	PS-02	200	3,200	IC06D31S
IB03514	PS-03	200	8,600	IC06D31S
1B03515	PS-04	200	7,400	IC06D31S
IB03516	PS-05	200	6,300	IC06D31S
IB03517	PS-06	200	3,100	IC06D31S
IB03518	PS-07	200	4,800	IC06D31S
1B03519	PS-08	200	940	IC06D31S
1B03520	PS-09	200	3,900	IC06D31S
1B03521	PS-10	0.40	N.D.	IC08D31S

Analytes reported as N.D. were not present at or above the reporting limit.



2852 Alton Ave., Irvine, CA 92606 1014 E. Cooley Dr., Suite A, Colton, CA 92324 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 9484 Chesapeake Dr., Suite 805, San Diego, CA 92123 9830 South 51st St., Suite B-120, Phoen (949) 261·1022 FAX (949) 261·1228 (909) 370·4667 FAX (909) 370·1046 (818) 779·1844 FAX (818) 779·1843 (619) 505·9596 FAX (619) 505·9689 (602) 785·0043 FAX (602) 785·0851

TRC Environmental Solutions 21 Technology Drive

21 Technology Drive Irvine, CA 92618 Attention: Richard Scott Client Project ID: 98-346

Lockheed

Sample Descript: Solid First Sample #: IB03522 Sampled: Feb 24, 1999 Received: Feb 25, 1999

Extracted: Mar 3-8, 1999
Analyzed: Mar 3-8, 1999

Reported: Mar 9, 1999

#### PERCHLORATE (EPA 300.0 Mod.)

Laboratory Number	Sample Description	Reporting Limit mg/Kg (ppm)	Sample Result mg/Kg (ppm)	QC Batch
1B03522	PS-11	200	7,400	IC06D31S
1B03523	PS-12	200	540	IC06D31S
IB03524	PS-13	1.0	13	IC07D31S
1B03525	PS-14	200	8,100	IC06D31S
IB03526	PS-15	200	5,300	IC06D31S
IB03527	PS-16	200	7,300	IC03D21S
1B03528	PS-17	0.40	N.D.	IC08D31S
IB03529	PS-18	400	600	IC03D21S
IB03530	PS-19	200	2,500	IC03D21S
IB03531	PS-20	200	5,400	IC03D21S

Analytes reported as N.D. were not present at or above the reporting limit.



2852 Alton Ave., Irvine, CA 92606 1014 E Cooley Dr., Suite A, Colton, CA 92324 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 9484 Chesapeake Dr., Suite 805, San Diego, CA 92123 9830 South 51st St., Suite B-120, Phoenix, AZ 85044 (949) 261-1022 FAX (949) 261-1228 (909) 370-4667 FAX (909) 370-1046 (818) 779-1844 FAX (818) 779-1843 (619) 505-9596 FAX (619) 505-9689 (602) 785-0043 FAX (602) 785-0851

TRC Environmental Solutions
21 Technology Drive

21 Technology Drive Irvine, CA 92618 Attention: Richard Scott Client Project ID: 98-346

Lockheed

Sample Descript: Solid First Sample #: IB03532

Sampled: Feb 24, 1999 Received: Feb 25, 1999

Extracted: Mar 3-6, 1999 Analyzed: Mar 3-6, 1999

Reported: Mar 9, 1999

#### PERCHLORATE (EPA 300.0 Mod.)

Laboratory Number	Sample Description	Reporting Limit mg/Kg (ppm)	Sample Result mg/Kg (ppm)	QC Batch
IB03532	PS-21	200	2,400	IC03D21S
1B03533	PS-22	200	1,300	IC03D21S
1B03534	PS-23	200	360	IC03D21S
1B03535	PS-24	200	2,400	IC03D21S
IB03536	PS-25	200	4,200	IC03D21S
IB03537	PS-26	200	3,000	IC03D21S
IB03538	PS-27	400	5,300	IC06D31S
1B03539	PS-28	200	2,500	IC03D21S
IB03551	PS-29	200	4,900	IC03D21S
IB03552	PS-30	400	13,000	IC06D31S

Analytes reported as N.D. were not present at or above the reporting limit.



2852 Alton Ave., Irvine, CA 92606 1014 E. Cooley Dr., Suite A, Colton, CA 92324 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 9484 Chesaoeake Dr., Suite 805, San Diego, CA 92123 9830 South 51st St., Suite B-120, Phoenix, AZ 85044 (949) 261-1022 FAX (949) 261-1228 (909) 370-4667 FAX (909) 370-1046 (818) 779-1844 FAX (818) 779-1843 (619) 505-9596 FAX (619) 505-9689 (602) 785-0043 FAX (602) 785-0851

TRC Environmental Solutions 21 Technology Drive Irvine, CA 92618

Attention: Richard Scott

Client Project ID: 98-346

Lockheed

Sample Descript: Solid First Sample #: IB03553 Sampled: Feb 24, 1999 Received: Feb 25, 1999 Extracted: Mar 3-6, 1999

Analyzed: Mar 3-6, 1999 Reported: Mar 9, 1999

#### PERCHLORATE (EPA 300.0 Mod.)

Laboratory Number	Sample Description	Reporting Limit mg/Kg (ppm)	Sample Result mg/Kg (ppm)	QC Batch
IB03553	PS-31	400	12,000	IC06D31S
1B03554	PS-32	200	6,300	IC03D21S
IB03555	PS-33	200	7,100	IC03D21S

Analytes reported as N.D. were not present at or above the reporting limit.



2852 Alton Ave., Irvine, CA 92606 1014 E. Cooley Dr., Suite A. Colton, CA 92324 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 9484 Chesapeake Dr., Suite 805, San Diego, CA 92123 9830 South 51st St., Suite B-120, Phoens, AZ 85044 (949) 261-1022 FAX (949) 261-1228 (909) 370-4667 FAX (909) 370-1046 (818) 779-1844 FAX (818) 779-1843 (619) 505-9596 FAX (619) 505-9689 (602) 785-0043 FAX (602) 785-0851

TRC Environmental Solutions 21 Technology Drive Irvine, CA 92618 Attention: Richard Scott

Method Blank

Extracted: Analyzed:

Mar 3-8, 1999 Mar 3-8, 1999

Reported:

Mar 3-8, 1999 Mar 9, 1999

#### PERCHLORATE (EPA 300.0 Mod.)

Sample Description	Reporting Limit mg/Kg (ppm)	Sample Result mg/Kg (ppm)	QC Batch
Method Blank	0.040	N.D.	IC03D21S
Method Blank	0.040	N.D.	IC06D31S
Method Blank	0.040	N.D.	IC07D31S
Method Blank	0.040	N.D.	IC08D31S

Analytes reported as N.D. were not present at or above the reporting limit.

2852 Alton Ave., Irvine, CA 92605 1014 E. Cooley Dr., Suite A. Colton, CA 92324 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 9484 Chesapeake Dr., Suite 805. San Diego, CA 92123 9830 South 51st St., Suite B-120, Phoenix, AZ 85044 (949) 261-1022 FAX (949) 261-1228 (909) 370-4667 FAX (909) 370-1046 (818) 779-1844 FAX (818) 779-1843 (619) 505-9596 FAX (619) 505-9689 (602) 785-0043 FAX (602) 785-0851

#### MS/MSD DATA REPORT

EPA METHOD: 300.0 Mod.

Matrix: Soil

**Date** 

Analyzed: 3/3/99

Sample:

IB03529

Batch:

IC03D21S

Analyte

R1

ppb

Sp MS

ppb

ppb

MSD

ppb

PR1 P

PR2

R2 RPD

PR Lir

RPD

MEAN

Limits

MPR

Acceptance

Perchlorate

598000 | 10000000 | 10900000 | 10900000 | 103% | 103% | 0.0% | 103% | 20 | 75-125

#### **Definition of Terms:**

R1..... Result of Sample Analysis

Sp..... Spike Concentration Added to Sample

MS..... Matrix Spike Result

MSD..... Matrix Spike Duplicate Result

RPD...... Relative Percent Difference; ((MS-MSD)/(MS+MSD)/2) X 100

Acceptance Limits. Statistically determined on an annual basis.

2852 Alton Ave., Irvine, CA 92606 1014 E. Cooley Dr., Suite A, Colton, CA 92324 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 9484 Chesapeake Dr., Suite 805, San Diego, CA 92123 9830 South 51st St., Suite B-120, Phoenix, AZ 85044

(949) 261-1022 FAX (949) 261-1228 (909) 370-4667 FAX (909) 370-1046 (818) 779-1844 FAX (818) 779-1843 (619) 505-9596 FAX (619) 505-9689 (602) 785-0043 FAX (602) 785-0851

#### MS/MSD DATA REPORT

EPA METHOD: 300.0 Mod.

Matrix: Soil

**Date** 

Analyzed: 3/6/99

Sample:

IB03512

Batch:

IC06D31S

Analyte	R1	Sp	MS	MSD	PR1	PR2	RPD	MEAN PR		ptance nits	
<del></del>	ppb	ppb	ppb	ppb	%	-%	%	%	RPD	MPR	
Perchlorate	4700000	10000000	15300000	15100000	106%	104%	1.3%	105%	20	75-125	i

#### **Definition of Terms:**

R1..... Result of Sample Analysis

Sp..... Spike Concentration Added to Sample

MS..... Matrix Spike Result

MSD..... Matrix Spike Duplicate Result

PR1......Percent Recovery of MS; ((MS-R1) / SP) X 100
PR2.....Percent Recovery of MSD; ((MSD-R1) / SP) X 100

RPD...... Relative Percent Difference; ((MS-MSD)/(MS+MSD)/2) X 100

Acceptance Limits. Statistically determined on an annual basis.



1014 E. Cooley Dr., Suite A. Colton. CA 92324 (909) 370-4667 FAX (909) 370-1046 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 9484 Chesapeake Dr., Suite 805. San Diego. CA 92123 9830 South 51st St., Suite B-120. Phoenix. AZ 85044

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#### MS/MSD DATA REPORT

EPA METHOD: 300.0 Mod.

Matrix: Soil

Date

Analyzed: 3/7/99

Sample:

IB03521

Batch:

IC07D31S

Analyte	R1	Sp	MS	MSD	PR1	PR2	RPD	MEAN PR		ptance nits
<del></del>	ppb	ppb	ppb	ppb	%	%	%	%	RPD	MPR
Perchlorate	0	1000000	963000	944000	96%	94%	2.0%	95%	20	75-125

## **Definition of Terms:**

R1..... Result of Sample Analysis

Sp..... Spike Concentration Added to Sample

MS..... Matrix Spike Result

MSD..... Matrix Spike Duplicate Result

PR1..... Percent Recovery of MS; ((MS-R1) / SP) X 100 PR2..... Percent Recovery of MSD; ((MSD-R1) / SP) X 100

RPD...... Relative Percent Difference; ((MS-MSD)/(MS+MSD)/2) X 100

Acceptance Limits. Statistically determined on an annual basis.



# Del Mar Analytical

2852 Alton Ave., Irvine, CA 92606 1014 E. Cooley Dr., Suite A, Colton, CA 92324 16525 Sherman Way, Suite C-11, Van Nuys. CA 91406 9484 Chesapeake Dr., Suite 805, San Diego, CA 92123 9830 South 51st St., Suite B-120, Phoenix, AZ 85044

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#### MS/MSD DATA REPORT

EPA METHOD: 300.0 Mod.

Matrix: Soil

Date

Analyzed:

3/8/99

Sample:

IB03521

Batch:

IC08D31S

Analyte

R1 Sp

ppb

ppb

MSD MS

ppb

ppb

PR1

%

PR2

%

**RPD** 

%

MEAN

PR

%

Acceptance

RPD

Limits

MPR

Perchlorate

100 90.1 90.1 90% 90% 0% 90% 20 75-125

#### **Definition of Terms:**

R1...... Result of Sample Analysis

Sp...... Spike Concentration Added to Sample

MS..... Matrix Spike Result

MSD..... Matrix Spike Duplicate Result

PR1..... Percent Recovery of MS; ((MS-R1) / SP) X 100

PR2..... Percent Recovery of MSD; ((MSD-R1) / SP) X 100

RPD...... Relative Percent Difference; ((MS-MSD)/(MS+MSD)/2) X 100

Acceptance Limits. . . . Statistically determined on an annual basis.

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2852 Alton Ave., frvine, CA 92606 1014 E. Cooley Dr., Suite A, Colton, CA 92324 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 9484 Chesapeake Dr., Suite 805, San Diego, CA 92123 9830 South 51st St., Suite B-120, Phoenix, AZ 85044 (949) 261-1022 FAX (949) 261-1228 (909) 370-4667 FAX (909) 370-1046 (818) 779-1844 FAX (818, 779-1843 (619) 505-9596 FAX (619) 505-9689 (602) 785-0043 FAX (602) 725-0851

#### LABORATORY REPORT

Prepared For:

TRC Environmental Solutions

21 Technology Drive Irvine, CA 92618

Attention:

Richard Scott

Project: 98-346

Lockheed

Sampled:

2/18/99

Received:

2/26/99

Reported:

3/9/99

This laboratory report is confidential and is intended for the sole use of Del Mar Analytical and its client. This entire report was reviewed and approved for release.

CA ELAP Certificate #1197 AZ DHS Licence #AZ0428

**DEL MAR ANALYTICAL** 

Fred Haley
Project Manager

Results pertain only to samples tested in the laboratory. This report shall not be reproduced, except in full, without written permission from Del Mar Analytical.

IB03793.TRC <1 of 3>



# Del Mar Analytical

2852 Alton Ave., Irvine, CA 92606 1014 E. Cooley Dr., Suite A, Colton, CA 92324 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 9484 Chesapeake Dr., Suite 805, San Diego, CA 92123 9830 South 51st St., Suite B-120, Phoenix, AZ 85044 (949) 261-1022 FAX (949) 261-1228 (909) 370-4667 FAX (909) 370-1046 (818) 779-1844 FAX (818) 779-1843 (619) 505-9596 FAX (619) 505-9689 (602) 785-0043 FAX (602) 785-0851

TRC Environmental Solutions
21 Technology Drive

Irvine, CA 92618
Attention: Richard Scott

Client Project ID: 98-346 Lockheed

Sample Descript: Solid First Sample #: IB03793 QC Batch: IC09D31S Sampled: Feb 18, 1999
Received: Feb 26, 1999
Extracted: Mar 9, 1999
Analyzed: Mar 9, 1999
Reported: Mar 9, 1999

#### PERCHLORATE (EPA 300.0 Mod.)

Laboratory Number	Sample Description	Reporting Limit mg/Kg (ppm)	Sample Result mg/Kg (ppm)
IB03793	PS-34	4,000	30,000

Analytes reported as N.D. were not present at or above the reporting limit.



2852 Alton Ave., Irvíne, CA 92606 1014 E. Cooley Dr., Suite A, Colton, CA 92324 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 9484 Chesapeake Dr., Suite 805, San Diego, CA 92123 9830 South 51st St., Suite B-120, Phoenix, AZ 85044 (949) 261-1022 FAX (949) 261-1228 (909) 370-4667 FAX (909) 370-1046 (818) 779-1844 FAX (818) 779-1843 (619) 505-9596 FAX (619) 505-9689 (602) 785-0043 FAX (602) 785-0851

TRC Environmental Solutions 21 Technology Drive Irvine, CA 92618 Attention: Richard Scott

Method Blank

Extracted: Analyzed:

Mar 9, 1999 Mar 9, 1999

Reported:

Mar 9, 1999

QC Batch: IC09D31S

#### PERCHLORATE (EPA 300.0 Mod.)

Sample Description	Reporting Limit	Sample Result
Description	mg/Kg	mg/Kg
	(ppm)	(ppm)
Method Blank	0.040	N.D.

Analytes reported as N.D. were not present at or above the reporting limit.



2852 Alton Äve.. Irvine. CA 92606 1314 E. Cooley Dr., Suite A. Colton. CA 92324 16525 Sherman Way, Suite C-11, Van Nuys. CA 91406 9484 Chesapeake Dr., Suite 805, San Diego. CA 92123 9630 South 51st St., Suite B-120, Phoenix. AZ 85044

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#### MS/MSD DATA REPORT

EPA METHOD: 300.0 Mod.

Matrix: Soil

Date

Analyzed: 3/9/99

Sample:

1B03793

Batch:

IC09D31S

Analyte		R1	Sp	MS	MSD	PR1	PR2	RPD	PR	Limi	
-	•	ppb	ppb	ppb	ppb	%	%	%	%	RPD	MPR

Perchlorate

9300000 | 500000000 | 524000000 | 530000000 | 99% | 100% | 1.1% | 100% | 20 | 75-125

#### **Definition of Terms:**

R1..... Result of Sample Analysis

Sp..... Spike Concentration Added to Sample

MS..... Matrix Spike Result

MSD..... Matrix Spike Duplicate Result

PR1..... Percent Recovery of MS; ((MS-R1) / SP) X 100
PR2.... Percent Recovery of MSD; ((MSD-R1) / SP) X 100

RPD..... Relative Percent Difference; ((MS-MSD)/(MS+MSD)/2) X 100

Acceptance Limits... Statistically determined on an annual basis.

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Total Number of	Samples:	Shipped:			r's Signa	iture:		<del>-</del> -																· · · · · · · · · · · · · · · · · · ·	
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Relinquished by	: YNE	Wife S	A THE					$\dashv$		K	9	2/	1/1	<u></u>								72	6-99		45 FM.
Received by:		The same	<u> </u>		·	·····		$\dashv$			V	=/	M41			<del></del>						126	144	2	5:40pm
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Special Instruction	ons / Shini	ment / Han	ndling/ Stor	age Requi	rements			L				<del>~</del>					7						TRC	.1	
Question	-		-	-			nord	. 5	Sco.	T						ď		M				21 Irvi	l Technology ne, Californi	a 9261	8
																	-						(949) 727-9	336	
property of	he material(s) listed are received for analysis and/or treatal roperty of the client and not TRC. At the conclusion of the naterial(s) will be returned to the client for eventual disposa							test	WOI	rk, a	ll re	main	ing	rmain the  2815 Mitchell Drive, Suite 103 Walnut Creek, California 94598 (925) 935-3294								e 103 94598			

A-Gen/Form (12/4//8/mms)

# APPENDIX A.2 AMERICAN PACIFIC CORPORATION

American Pacific Corporation - Utah Operations 10622 West 6400 North, Cedar City, Utah 84720 Tel: (435) 865-5000 Fax: (435) 865-5029

Laboratory				. Р	roject No.:	98-346 L	_ockheed
(if not AMP	AC)			Re	port Date:	March 2	22, 1999
Client:		TRC			Sample ID:	PS-02/	IB03513
		21 Technology Drive Irvine, CA 92618		. Re	ceipt Date:	March	12, 1999
Collection	Date:	February 24, 1	999	Collec	tion Time:	N	1A
Preservation	on:	Refrigeration	<u>1</u>	Prepara	ation Date:	March	16, 1999
		Т	est Resul	ts			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate  * MDL = Minimum De	3/19/99	WEC-LAB-060-WI-54	1000	ppb	349,800	1350	NA
Descriptio	n of Devia	ations and/or QC Failu	res:				
Authorized	d Signatu	re and Title:		Analytical	Labs and Pr	oduct Dev	elopment

# American Pacific Corporation - Utah Operations 10622 West 6400 North, Cedar City, Utah 84720 Tel: (435) 865-5000 Fax: (435) 865-5029

Laboratory	<b>/</b> :			. Pi	roject No.:	98-346 L	_ockheed
(if not AMP	AC)		· · · · · · · · · · · · · · · · · · ·	Re	port Date:	March 2	22, 1999
Client:		TRC		. 8	Sample ID:	PS-03/I	IB03514
		21 Technology Drive Irvine, CA 92618		Red	ceipt Date:	March 1	12, 1999
Collection	Date:	February 24, 19	999	Collec	tion Time:	N	IA
Preservati	on:	Refrigeration	າ	Prepara	ntion Date:	March 1	16, 1999
			· · · · · · · · · · · · · · · · · · ·				
			est Resul	1			T
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/19/99	WEC-LAB-060-WI-54	2500	ppb	895,800	1350	NA
Descriptio		ations and/or QC Failu	res:				
Authorize	d Signatuı	re and Title:	Director	Analytical	Labs and Pr	oduct Dev	elopment
				-			•

American Pacific Corporation - Utah Operations 10622 West 6400 North, Cedar City, Utah 84720 Tel: (435) 865-5000 Fax: (435) 865-5029

Laboratory				P	roject No.: _	98-346 1	Lockheed
(if not AMP	AC)	•		Re	eport Date:	March	22, 1999
Client:		TRC			Sample ID:	PS-04/	IB03515
		21 Technology Drive Irvine, CA 92618		Re	ceipt Date:	March	12, 1999
Collection	Date:	February 24, 1	999	Collec	tion Time:	1	NA
Preservati	on:	Refrigeration	<u> </u>	Prepara	ation Date: _	March	16, 1999
		T	est Result	s			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/19/99	WEC-LAB-060-WI-54	2500	ppb	762,500	1350	NA
Descriptio	n of Devia	ations and/or QC Failu	res:				
Authorize	d Signatu	re and Title:	الم كان المالية	رز ار Analytical	Cullin Labs and Pr	auoduct Dev	elopment

Laboratory			1	_ P	roject No.: _	98-346 L	ockheed
(if not AMP	AC)			- Re	port Date:	March 2	22, 1999
Client:		TRC		_	Sample ID:	PS-05/I	B03516
		21 Technology Drive Irvine, CA 92618		Rec	ceipt Date:	March 1	12, 1999
Collection	Date:	February 24, 1	999	Collec	tion Time:		IA
Preservati	on:	Refrigeration	<u> </u>	_ Prepara	ation Date:	March ·	16, 1999
		7	est Resul	ts		<del> </del>	
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/19/99	WEC-LAB-060-WI-54	2500	ppb	670,500	1350	NA
*MDL = Minimum De Descriptio		ations and/or QC Failu	res:				
Authorized	d Signatu	re and Title:	50 Directo	r Analytical	Rulu Labs and Pr	رزند_ oduct Dev	elopment

Laboratory: Project No.: 98-346 Lockheed (if not AMPAC) Report Date: March 22, 1999 Sample ID: PS-06/IB03517 Client: 21 Technology Drive Receipt Date: March 12, 1999 Irvine, CA 92618 **Collection Date:** February 24, 1999 Collection Time: Preparation Date: March 16, 1999 Preservation: Refrigeration Test Results Analyte Date MDL\* Units Result Analyst Deviations Method 3/19/99 322,000 1350 NA Perchlorate WEC-LAB-060-WI-54 1000 ppb MDL = Minimum Detectable Limit Description of Deviations and/or QC Failures: **Authorized Signature and Title:** 

Directór Analytical Labs and Product Development

Laboratory				Pı	roject No.: _	98-346 L	ockheed_
(if not AMP	AC)			Re	port Date:	March 2	22, 1999
Client:		TRC		. 8	Sample ID:	PS-07/I	B03518
		21 Technology Drive Irvine, CA 92618		Red	eipt Date: _	March 1	12, 1999
Collection	Date:	February 24, 1	999	Collec	tion Time:	1	IA
Preservati	on:	Refrigeration	1	Prepara	ation Date:	March 1	16, 1999
			est Result	s			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/19/99	WEC-LAB-060-WI-54	1000	ppb	473,900	1350	NA
Descriptio		tions and/or QC Failu	res:				
Authorize	d Signatui	e and Title:	Director	Analytical I	Labs and Pr	oduct Dev	elopment

Laboratory	<b>/</b> :			_ P	roject No.:_	98-346 L	_ockheed
(if not AMP	AC)			- Re	port Date:	March 2	22, 1999
Client:		TRC		_	Sample ID:	PS-08/I	B03519
		21 Technology Drive Irvine, CA 92618		Rec	ceipt Date:	March 1	12, 1999
Collection	Date:	February 24, 1	999	Collec	tion Time:	N	IA
Preservati	on:	Refrigeration	า	_ Prepara	ation Date:	March '	16, 1999
		•	est Resul	140			
		1					
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate  * MDL = Minimum De	3/17/99	WEC-LAB-060-WI-54	500	ppb	109,300	1350	NA
		ations and/or QC Failu	res:			, , , , , , , , , , , , , , , , , , ,	
Authorize	d Signatu	re and Title:		entru)	,	au	elonment
			Directo	i Allalylloal	Lans allu Fl	odaci Devi	Piohineur

Laboratory				_ P	roject No.: <sub>-</sub>	98-346 l	_ockheed
(if not AMP	AC)		· · · · · · · · · · · · · · · · · · ·	- Re	port Date:	March :	22, 1999
Client:		TRC	-	_	Sample ID:	PS-09/	IB03520
		21 Technology Drive Irvine, CA 92618	· · · · · · · · · · · · · · · · · · ·	Re	ceipt Date:	March	12, 1999
Collection	Date:	February 24, 1	999	Collec	tion Time:	1	NA
Preservati	on:	Refrigeration	1	Prepara	ation Date:	March	16, 1999
		T	est Resul	ts			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/19/99	WEC-LAB-060-WI-54	1000	ppb	438,200	1350	NA
• MDL = Minimum De  Descriptio		ations and/or QC Failu	res:				
				/ , `./	7 //		
Authorize	d Signatu	re and Title:	Directo	<i>الناحال</i> : r Analytical	Labs and Pr	oduct Dev	elopment

Laboratory	<b>/</b> :			_ Pi	roject No.: _	98-346 L	_ockheed
(if not AMP	AC)			- Re	port Date:	March 2	22, 1999
Client:		TRC		_	Sample ID:	PS-10/	IB03521
		21 Technology Drive Irvine, CA 92618		- Red	ceipt Date:	March	12, 1999
Collection	Date:	February 24, 1	999	Collec	tion Time:		IA
Preservati	on:	Refrigeration	1	Prepara	ation Date:	March	16, 1999
<del>4</del>		T	est Resul	ts			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/22/99	WEC-LAB-060-WI-54	60	ppb	< 60	1350	NA
Descriptio		ations and/or QC Failu	res:			·	
Authorized	d Signatu	re and Title:	Directo	r Analytical	Puli	lielu—	olonmont
		•	Directo	i Allalylical	Laus allu Pi	oduct Dev	erobineur

Laboratory	•			. Р	roject No.: _	98-346 L	_ockheed
(if not AMP	'AC)			Re	eport Date:	March 2	22, 1999
Client:		TRC		_	Sample ID:	PS-11/	IB03522
		21 Technology Drive Irvine, CA 92618		Re	ceipt Date:	March 1	12, 1999
Collection	Date:	February 24, 1	999	Collec	tion Time:		NA
Preservati	on:	Refrigeration	n	Prepara	ation Date:	March	16, 1999
		7	est Resul	s			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/19/99	WEC-LAB-060-WI-54	2500	ppb	816,600	1350	NA
*MDL = Minimum De Descriptio		ations and/or QC Failu	ires:				
Authorize	d Signatuı	e and Title:	_ Ki	A)	Dula	ar_	
			Directo	Analytical	Labs and Pr	oduct Dev	elopment

Laboratory				P	roject No.: _	98-346 i	Lockheed
(if not AMP	AC)			Re	port Date:	March :	22, 1999
Client:		TRC			Sample ID:	PS-12/	IB03523
		21 Technology Drive Irvine, CA 92618		Red	ceipt Date:	March	12, 1999
Collection	Date:	February 24, 19	999	Collec	tion Time:	1	NA
Preservati	on:	Refrigeration	1	Prepara	ation Date:	March	16, 1999
		Т	est Result	s			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/17/99	WEC-LAB-060-WI-54	500	ppb	63,640	1350	NA
Descriptio	n of Devia	ations and/or QC Failu	res:				
Authorize	d Signatu	re and Title:	#S Director	Analytical	) Rull Labs and Pr	ر مصد oduct Dev	elopment

Laborator	Laboratory: (if not AMPAC)			. Р	roject No.: _	98-346 I	_ockheed
(if not AMP	AC)			Re	port Date:	March :	22, 1999
Client:		TRC 21 Technology Drive			Sample ID:	PS-13/	IB03524
		Irvine, CA 92618		Red	ceipt Date:	March	12, 1999
Collection	Date:	February 24, 19	999	Collec	tion Time:		NA
Preservati	on:	Refrigeration	<u> </u>	Prepara	ation Date:	March	16, 1999
		T	est Result	s			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/22/99	WEC-LAB-060-WI-54	600	ppb	< 600	1350	NA
Descriptio	n of Devia	ations and/or QC Failu	res:				
Authorize	d Signatu	re and Title:	Director	Analytical	Dulin Labs and Pr	oduct Dev	elopment

Laboratory				. P	roject No.:	98-346 L	_ockheed
(if not AMP	AC)			Re	port Date:	March 2	22, 1999
Client:		TRC			Sample ID:	PS-14/	IB03525
		21 Technology Drive Irvine, CA 92618		Red	ceipt Date:	March '	12, 1999
Collection	Date:	February 24, 1	999	Collec	tion Time:	١	NA
Preservati	on:	Refrigeration	n	Prepara	ation Date:	March	16, 1999
			est Result	ts			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/19/99	WEC-LAB-060-WI-54	2500	ppb	948,300	1350	NA
Descriptio	n of Devia	ations and/or QC Failu	ires:				
Authorize	d Signatu	re and Title:	15 ca Director		Cullu Labs and Pr	ر oduct Dev	elopment

Laboratory				. Р	roject No.: _	98-346 L	_ockneea
(if not AMP	AC)			Re	port Date:	March 2	22, 1999
Client:		TRC			Sample ID: _	PS-15/	B03526
		21 Technology Drive Irvine, CA 92618	11.11.1.1.1.1.1.1.1.1.1.1.1.1.1.1.1.1.1.	Red	ceipt Date:	March '	12, 1999
Collection	Date:	February 24, 1	999	Collec	tion Time:		IA
Preservation	on:	Refrigeration	<u> </u>	. Prepara	ation Date:	March	16, 1999
		1	est Resul	is		· · · · · · · · · · · · · · · · · · ·	
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/19/99	WEC-LAB-060-WI-54	2500	ppb	606,500	1350	NA
• MDL = Minimum De  Descriptio		ations and/or QC Failu	res:			,	
Authorized	d Signatu	re and Title:		uk.	Rulin	<u>a-</u>	
			Director	Analytical	Labs and Pr	oduct Dev	elopment

Laboratory				P	roject No.: <sub>-</sub>	98-346 l	_ockheed
(if not AMP	AC)			Re	port Date:	March :	22, 1999
Client:		TRC			Sample ID:	PS-16/	IB03527
		21 Technology Drive Irvine, CA 92618		Re	ceipt Date:	March	12, 1999
Collection	Date:	February 24, 19	999	Collec	tion Time:		NA
Preservati	on:	Refrigeration	n	Prepara	ation Date:	March	16, 1999
		T	est Result	s			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/19/99	WEC-LAB-060-WI-54	2500	ppb	751,600	1350	NA
Descriptio	n of Devia	ations and/or QC Failu	res:				
Authorized	d Signatu	re and Title:	ク かと Director	Analytical	Labs and Pr	oduct Dev	elopment

Laboratory	<b>/</b> :			Pi	roject No.:	98-346 L	ockheed
(if not AMP.	AC)			Re	port Date:	March 2	22, 1999
Client:		TRC			Sample ID:	PS-17/I	B03528
		21 Technology Drive Irvine, CA 92618		Red	eipt Date:	March 1	12, 1999
Collection	Date:	February 24, 19	999	Collec	tion Time:	N	IA
Preservation	on:	Refrigeration	1	Prepara	ation Date:	March '	16, 1999
-			est Result	s			
Analyte	Date	Method	MDL*	MDL* Units Result		Analyst	Deviations
Perchlorate	3/22/99	T			Result < 600	Analyst 1350	Deviations NA
Perchlorate • MDL = Minimum Del	3/22/99 tectable Limit	Method	MDL* 600	Units			<del> </del>

Laboratory			Ju 122	P	roject No.:_	98-346 [	ockheed
(if not AMP	AC)			Re	port Date:	March :	22, 1999
Client:		TRC			Sample ID:	PS-18/	IB03529
Irvine, CA 92618		21 Technology Drive Irvine, CA 92618			March 12, 1999		
Collection	Date:	February 24, 1	999	Collec	tion Time:		1A
Preservation: Refrige		Refrigeration	<u> </u>	Prepara	ation Date:	March	16, 1999
		Т	est Result	s			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/17/99	WEC-LAB-060-WI-54	500	ppb	73,300	1350	NA
Descriptio		ations and/or QC Failu	res:				
Authorize	d Signatu	re and Title:		(ku)	Rulu	a	
			Director	Analytical	Labs and Pr	oduct Dev	elopment

Laboratory				. Р	roject No.: _	98-346 l	_ockheed
(if not AMP	AC)			. Re	port Date:	March :	22, 1999
Client:		TRC			Sample ID:	PS-19/	IB03530
		21 Technology Drive Irvine, CA 92618		Receipt Date:		March 12, 1999	
Collection Date: February 24,			999	Collec	tion Time:	NA	
Preservation: Refrigeration		1	Preparation Date:			March 16, 1999	
		T	est Resul	ts			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/17/99	WEC-LAB-060-WI-54	500	ppb	265,400	1350	NA
*MDL = Minimum De Descriptio		ations and/or QC Failu	res:				
Authorized	d Signatu	re and Title:	Director	Analytical	Labs and Pr	oduct Dev	elopment

Laboratory				. Pi	roject No.: _	98-346 L	_ockheed
(if not AMP	AC)			Re	port Date:	March 2	22, 1999
Client:		TRC	,		Sample ID: _	PS-20/	IB03531
		21 Technology Drive Irvine, CA 92618		Red	eipt Date:	March 12, 1999	
Collection	Date:	February 24, 1	999	Collec	tion Time:		IA
Preservation: Refrige		Refrigeration	1	Prepara	ation Date:	March	16, 1999
		T	est Result	s			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/19/99	WEC-LAB-060-WI-54	1000	ppb	538,600	1350	NA
*MDL = Minimum De  Descriptio		ations and/or QC Failu	res:				
Authorize	d Signatu	re and Title:		Analytical	Duling Labs and Pro	مر oduct Dev	elopment

Laboratory:(if not AMPAC)			. P	roject No.:	98-346 [	_ockheed	
(II NOL AIVII	۸٥)			Re	port Date:	March 2	22, 1999
Client:		TRC 21 Technology Drive			Sample ID:	PS-21/	IB03532
		Irvine, CA 92618		Red	ceipt Date:	March 12, 1999	
Collection	Date:	February 24, 1	999	Collec	tion Time:		NA
Preservation: Refrigera		Refrigeration	<u>n</u>	Prepara	ation Date:	March	16, 1999
		1	est Result	S		·	
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/17/99	WEC-LAB-060-WI-54	500	ppb	296,000	1350	NA
		ations and/or QC Failu	res:				
Authorize	d Signatuı	re and Title:	Director	Analytical	Quhi. Labs and Pr	oduct Dev	elopment

	<b>y:</b>			. г	roject No.: _	30-340 [	_ockneea
(if not AMP	(if not AMPAC)			Re	port Date: _	March 22, 1999	
Client:		TRC		. ;	Sample ID:	PS-22/	IB03533
	21 Technology Irvine, CA 926		Receipt Date:		March 12, 1999		
Collection	Date:	February 24, 1	999	Collec	tion Time:	1	NA
Preservation: Refriger			n	Prepara	ation Date:	March	16, 1999
		Т	est Result	s			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate * MDL = Minimum De	3/17/99 etectable Limit	WEC-LAB-060-WI-54	500	ppb	154,900	1350	NA
* MDL = Minimum De	etectable Limit	WEC-LAB-060-WI-54		ppb	154,900	1350	NA NA

Laboratory				. Р	roject No.:	98-346 t	_ockheed
(if not AMP	AC)			Re	eport Date:	March :	22, 1999
Client:		TRC			Sample ID:	PS-23/	IB03534
		21 Technology Drive Irvine, CA 92618		. Re	ceipt Date:	March 12, 1999	
Collection Date: February 2  Preservation: Refriger			999	Collec	tion Time:	NA	
Preservation: Refriger		Refrigeration	n	Preparation Date:		March 16, 1999	
		Т	est Result	ts		1	
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/17/99	WEC-LAB-060-WI-54	500	ppb	46,460	1350	NA
*MDL = Minimum De Descriptio		itions and/or QC Failu	res:				
Authorize	d Signatu	e and Title:	H u	Analytical	Labs and Pr	oduct Dev	elopment

Laboratory	<b>/:</b>			P	roject No.:	98-346 L	_ockheed
(if not AMP	AC)			Report Date:		March 22, 1999	
Client:		TRC			Sample ID:	PS-24/	IB03535
	21 Technology Drive Irvine, CA 92618			Receipt Date:		March 12, 1999	
Collection Date: February 2			999	_ Collection Time:		NA	
Preservation: Refriger		Refrigeration	}	Preparation Date:		March 16, 1999	
		т	est Result	·e		· · · · · · · · · · · · · · · · · · ·	
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchiorate	3/17/99	WEC-LAB-060-WI-54	500	ppb	259,200	1350	NA
Descriptio		ations and/or QC Failu	res:				
Authorized	1 Signatu	re and Title:	-رو	Sintra	) D. C.		
Additionized	a Oignatu	ic and fille.	Director	/	Labs and Pr	oduct Dev	elopment

Laboratory				. Р	roject No.: _	98-346 l	_ockheed
(if not AMP	AC)			- Re	port Date:	March 2	22, 1999
Client:		TRC		_ ;	Sample ID:	PS-25/	IB03536
		21 Technology Drive Irvine, CA 92618		Re	ceipt Date:	March	12, 1999
Collection	Date:	February 24, 1	999	Collec	tion Time:		NA
Preservation: Refriger		Refrigeration	<u>1</u>	Prepara	ation Date:	March	16, 1999
		Τ	est Resul	ts			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate * MDL = Minimum De	3/19/99	WEC-LAB-060-WI-54	1000	ppb	422,300	1350	NA
Descriptio	n of Devia	ations and/or QC Failu	res:				
Authorize	d Signatu	re and Title:	Director	ال مار Analytical	Cuki_ Labs and Pr	വ oduct Dev	elopment

Laboratory	<b>/</b> :			Pi	roject No.: _	98-346 L	.ockheed
(if not AMP	AC)			Re	port Date:	March 2	22, 1999
Client:		TRC			Sample ID:	PS-26/I	B03537
	21 Technology Drive Irvine, CA 92618			Receipt Date:		March 12, 1999	
Collection Date: February 24			999	Collection Time:		NA	
Preservation: Refrigera		Refrigeration	1	_ Preparation Date:		March 16, 1999	
			est Result	s			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/17/99	WEC-LAB-060-WI-54	500	ppb	307,300	1350	NA
Descriptio		tions and/or QC Failu	res:				
Authorize	d Signatur	re and Title:	Director	Analytical I	Labs and Pro	oduct Deve	elopment

			. P	roject No.:_	90-340 1	<u>-ockneed</u>
			Re	port Date:	March :	22, 1999
	TRC			Sample ID:	PS-27/	IB03538
	21 Technology Drive Irvine, CA 92618		Red	ceipt Date:	March	12, 1999
:	February 24, 1	999	Collec	tion Time:		NA
Preservation: Refrigerat		n	Prepara	ation Date:	March	16, 1999
	7	Test Result	s			
ate	Method	MDL*	Units	Result	Analyst	Deviations
9/99	WEC-LAB-060-WI-54	2500	ppb	668,200	1350	NA
Devia	tions and/or QC Failu	ıres:				
···						
	ate 9/99 imit Devia	21 Technology Drive Irvine, CA 92618  February 24, 1  Refrigeratio  ate Method 9/99 WEC-LAB-060-WI-54	21 Technology Drive Irvine, CA 92618  February 24, 1999  Refrigeration  Test Result  ate Method MDL*  9/99 WEC-LAB-060-WI-54 2500	TRC 21 Technology Drive Irvine, CA 92618 Reconstruction Preparation Preparation Preparation Preparation Websites Website	TRC Sample ID:  21 Technology Drive Irvine, CA 92618 Receipt Date:  February 24, 1999 Collection Time:  Refrigeration Preparation Date:  Test Results  ate Method MDL* Units Result  9/99 WEC-LAB-060-WI-54 2500 ppb 668,200	21 Technology Drive

Laboratory	•			. P	roject No.: _	98-346 [	_ockneea
(if not AMP	AC)		· · · · · · · · · · · · · · · · · · ·	Re	port Date:	March :	22, 1999
Client:		TRC			Sample ID:	PS-28/	IB03539
	21 Technology Drive Irvine, CA 92618			Receipt Date:		March 12, 1999	
Collection	Date:	February 24, 1	999	Collec	tion Time:		1A
Preservation: Refrigerati		<u>1</u>	Prepara	ation Date:	March	16, 1999	
		ī	est Result				<del></del>
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/17/99	WEC-LAB-060-WI-54	500	ppb	259,900	1350	NA
* MDL = Minimum De  Descriptio		ations and/or QC Failu	res:				
Authorize	d Signatu	re and Title:		1. tw	Ruli	-dd-D	-1
			Director	Analytical	Labs and Pr	oduct Dev	elopment

Laboratory: (if not AMPAC)				P	roject No.:	98-346 L	ockheed
(II NOT AIMP	AC)			Re	port Date:	March 2	22, 1999
Client:		TRC			Sample ID:	PS-29/	IB03551
		21 Technology Drive Irvine, CA 92618		Red	ceipt Date:	March	12, 1999
Collection	Date:	February 24, 19	999	Collec	tion Time:	N	JA
Preservation: Refrigerat		Refrigeration	1	Prepara	ation Date:	March	16, 1999
	<del></del>	T	est Result	s			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations
Perchlorate	3/19/99	WEC-LAB-060-WI-54	1000	ppb	518,900	1350	NA
Descriptio		ations and/or QC Failu	res:				
Authorize	d Signatu	re and Title:		Analytical	Rulli Labs and Pr	oduct Dev	elopment

Laboratory				. Р	roject No.: _	98-346 l	_ockheed	
(if not AMP	AC)			. Re	eport Date:	March 22, 1999		
Client:		TRC 21 Technology Drive		-	Sample ID: _	PS-30/IB03552		
		Irvine, CA 92618		Re	ceipt Date:	March 12, 1999		
Collection	Date:	February 24, 19	999	Collec	tion Time:	NA		
Preservati	on:	Refrigeration	1	Prepar	ation Date:	March 16, 1999		
		Т	est Resul	s				
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations	
Perchiorate	3/19/99	WEC-LAB-060-WI-54	2500	ppb	1,430,000	1350	NA	
*MDL = Minmum De Descriptio		ations and/or QC Failu	res:					
Authorized	d Signatu	re and Title:	Director	ساسا Analytical	Cultus Labs and Pr	oduct Dev	elopment	

Laboratory				P	roject No.: _	98-346 L	.ockheed	
(if not AMP	AC)			Re	port Date:	March 22, 1999		
Client:		TRC	· · · · · · · · · · · · · · · · · · ·		Sample ID:	PS-31/IB03553		
		21 Technology Drive Irvine, CA 92618		Red	ceipt Date:	March 12, 1999		
Collection	Date:	February 24, 1	999	Collec	tion Time:	N	IA	
Preservati	on:	Refrigeration	ı	Prepara	ation Date:	March 1	16, 1999	
		Т	est Result	s				
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations	
Perchlorate	3/19/99	WEC-LAB-060-WI-54	2500	ppb	1,329,000	1350	NA	
• MDL = Minimum De Descriptio		ations and/or QC Failu	res:					
Authorize	d Signatuı	re and Title:	**************************************	J. W.	Pula.			
			Director	Analytical	Labs and Pr	oduct Deve	elopment	

Laboratory	<b>/</b> :			. P	roject No.: _	98-346 l	_ockheed	
(if not AMP	AC)			Re	eport Date:	March 22, 1999		
Client:		TRC			Sample ID: _	PS-32/IB03554		
		21 Technology Drive Irvine, CA 92618		Re	ceipt Date: _	March 12, 1999		
Collection	Date:	February 24, 1	999	Collec	ction Time:	NA		
Preservation	on:	Refrigeration	1	Prepara	ation Date:	March 16, 1999		
			est Resul	ts	······································			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations	
Perchlorate	3/19/99	WEC-LAB-060-WI-54	2500	ppb	638,100	1350	NA	
* MDL = Minimum De  Descriptio		ations and/or QC Failu	res:					
Authorized	d Signatu	re and Title:	Director	(Applytical	Richard Pr	educt Day	olonmont	

Laboratory				. P	roject No.: _	98-346 Lockheed		
(if not AMP	AC)			. Re	port Date:	March 22, 1999		
Client:		TRC		. 8	Sample ID:	PS-33/IB03555		
		21 Technology Drive Irvine, CA 92618		Red	ceipt Date:	March 12, 1999		
Collection	Date:	February 24, 1	999	. Collec	tion Time:		NA	
Preservation:		Refrigeration	n	Prepara	ation Date:	March 16, 1999		
	· • • • • • • • • • • • • • • • • •	1	est Result	ts				
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations	
Perchlorate	3/19/99	WEC-LAB-060-WI-54	2500	ppb	756,300	1350	NA	
Descriptio	n of Devia	ations and/or QC Failu	ires:					
Authorized	d Signatu	re and Title:		Analytical	Oulu Labs and Pr	oduct Dev	elopment	

Laboratory				. Р	roject No.: _	98-346 L	_ockheed	
(if not AMP	AC)			Re	eport Date:	March 2	22, 1999	
Client:		TRC			Sample ID:	PS-34/IB03793		
		21 Technology Drive Irvine, CA 92618		Re	ceipt Date:	March 12, 1999		
Collection	Date:	February 18, 19	999	Collec	tion Time:		NA	
Preservation	on:	Refrigeration	1	Prepara	ation Date:	March 16, 1999		
-		Т	est Resul	ts				
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations	
Perchiorate	3/19/99	WEC-LAB-060-WI-54	5000	ppb	2,631,000	1350	NA	
Descriptio	n of Devia	ations and/or QC Failu	res:					
Authorized	d Signatu	re and Title:	F 20 Director	ال يا Analytical	Labs and Pro	oduct Dev	elopment	

Laboratory	<b>/</b> :			. Р	roject No.: ˌ	98-346 L	_ockheed	
(if not AMP	AC)			Re	eport Date:	March 2	22, 1999	
Client:		TRC		. :	Sample ID:	Bla	ank	
		21 Technology Drive Irvine, CA 92618		Re	ceipt Date:	March 12, 1999		
Collection	Date:	NA		Collec	tion Time:		JA .	
Preservati	on:	Refrigeration	າ	Prepara	ation Date:	March 16, 1999		
		Ţ	est Result	ts				
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations	
Perchlorate	3/22/99	WEC-LAB-060-WI-54	3	ppb	< 3	1350	NA	
Descriptio		ations and/or QC Failu	res:					
				, , ,				
Authorize	d Signatu	re and Title:	Ke	uku	Rule	سار ا		
	_	•	Director	Analytical	Labs and Pr	oduct Dev	elopment	

Laboratory	•			. Р	roject No.: _	98-346	Lockheed	
(if not AMP	AC)			Re	port Date:	March 22, 1999		
Client:		TRC			Sample ID: _	LCS1		
		21 Technology Drive Irvine, CA 92618		Red	ceipt Date:	March 12, 1999		
Collection	Date:	NA		Collec	tion Time:	NA		
Preservati	on:	Refrigeration	<u>1</u>	Prepara	ation Date:	March 16, 1999		
		Т	est Resul	s	· · · · · · · · · · · · · · · · · · ·			
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations	
Perchlorate	3/22/99	WEC-LAB-060-WI-54	3	ppb	96	1350	NA	
Descriptio	n of Devia	itions and/or QC Failu	res:					
Authorize	d Signatu	re and Title:	ار Director	Analytical	Labs and Pr	oduct Dev	elopment	

Laborator				. P	roject No.: _	98-346 1	Lockheed	
(if not AMF	AC)			- Re	eport Date:	March 22, 1999		
Client:		TRC		_	Sample ID:	LCS2		
		21 Technology Drive Irvine, CA 92618		- Re	ceipt Date:	March 12, 1999		
Collection	Date:	NA		Collec	tion Time:		٧A	
Preservati	on:	Refrigeration	n	Prepara	ation Date:	March 16, 1999		
		T	est Result	ts				
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations	
Perchlorate	3/22/99	WEC-LAB-060-WI-54	3	ppb	104	1350	NA	
Descriptio	on of Devia	ations and/or QC Failu	res:					
Authoriza	d Cianatu	ro and Title.		la ) 6	0 0			
Authorize	u əigilatu	re and Title:	Director		Labs and Pr	oduct Dev	elopment	

	Del <b>P</b> rAnalytical
~	2852 Alton Sun Indos CA 82808 /5681 261-1022

FAX (945) 261-1228 1814 E. Cooley Dr., Sulta A, Colton, CA 92324 (909) 370-4667 FAX (909) 370-1048

### To: American Pacific Corporation

16525 Sherman Way, Suite C-11, Van Nuya, CA 91406 (618) 779-1844 FAX (818) 779-1843 CHAIN OF CUSTODY FORM Attn: Lairy Commings Page 1 of 3 2465 W 12th St., Suite 1, Temps, AZ 85281 (602) 968-8272 FAX (602) 968-1336 9484 Cheespeake Dr., Suite 806, San Diego, CA 92123 (619) 605-9596 FAX (619) 506-9689 Client Name/Address: Project/PO Number: **Analysis Required** 98-346 zi Technology Drive Irvine, CA 92618 H/TUP: 3/24 Project Manager: Richard Scott (949) 727-9336 # of Sampling Preservatives Sample Sample i Container Description Matrix Type Cont. Date/Time Special Instructions \* extract PS-01/IB03512 Glass PS-02 /1803513 PS-03 /1803514 PS-04/1803515 75-05 /JB03516 PS-06/IB03517 DS07/IB035/8 DS-08/IB03519 PS-09 / IB03520 15-10 /IB03521 PS-11/IB03522 PS-12/IB03523 PS-13 /IB03524 Date /Time: // Date /Time: Relinguished By: Received by: Turnaround Time: (Check) Fed-EX same day 72 hours \_\_\_\_\_ Relinquished By: 24 hours 48 hours normal Relinquished By: Date /Time: Sample Integrity: (Check) intact on ice

Note: By relinquishing samples to Del Mar Analytical, client agrees to pay for the services requested on this chain of custody form and any additional analyses performed on this project. Payment for services is due within 30 days from the date of invoice. Sample(s) will be disposed of after 30 days.

re., irvine, CA 92806 (949) 261-1022 FAX (848) 261-1228 1014 E. Cooley Dr., Suite A, Colton, CA 92324 (909) 570-4667 FAX (909) 370-1046 18525 Sherman Way, Suite C-11, Van Huya, CA \$1406 (818) 779-1844 FAX (818) 779-1843 To:

### CHAIN OF CUSTODY FORM

2465 W 12th St., Sulfo 1, Tempa, AZ 85281 (802) 968-8272 FAX (802) 968-1338 CHAIN OF CUSTODY FORM  Page																	
Client Name/Address:			1 .	PO Numbe						Ana	lysis Req	uired		<del></del>		<del>'',                                   </del>	
ZITECHNOLOGY D IVING, CA G	114e 1261	8	98 Lc	-341 XKe	s e	d										Mtx=	extrac
Client Name/Address:  21 Technology I  Zivine, CA G  Project Manager:  Pichard Scot  Sampler:	-+		Phone I	Number: mber:													
Sample Description	Preservatives										Specia	Instructions					
PS-29   IB03551	5*	Glass	1	224	199				 	<u> </u>	ļ					HITUF	3/24
PS-30/ IB03552	7											<u> </u>					1
PS-31/IB03553															<u> </u>		
PS-32/IB03554												<u> </u>		<u> </u>			
PS-33/IB03555				1 ×								<u> </u>			<u> </u>		<i>b</i>
PS-34/IB03743	1	4		2:/18	199	*										X H/T	31/8 gu
Blank		Plustic														_	
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LCS2 100 ppb		4	4														
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Relinquished By:		Date /Tinge				Pageined by		<u> </u>	Date /Ti	me.			T			hash	
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Relinquished By:		Date /Time:				Received by:			Date /Tir	ne:			24 hou			5 days normal	
Relinquished By:		Date /Time:				Received in Lat	b by: Date /Time: Sample Integrity: (Check)										

Note: By relinquishing samples to Del Mar Analytical, client agrees to pay for the services requested on this chain of custody form and any additional analyses performed on this project. Payment for services is due within 30 days from the date of invoice. Sample(s) will be disposed of after 30 days.

2852 Alfon Ave., Irvine, CA 92606 (949) 261-1022 FAX (949) 261-1228
1014 E. Cooley Dr., Suite A, Colton, CA 92324 (909) 370-4867 FAX (909) 370-1048
16325 Sherman Way, Suite C-11, Van Huys, CA 91406 (618) 779-1844 FAX (618) 779-1843
2465 W 12th St., Suite 1, Tempe, AZ 85261 (602) 968-8272 FAX (602) 968-1338

### CHAIN OF CUSTODY FORM

4.71.3

Page 2 of 3

Client Name/Address: Project/PO Number:									, ug								
Client Name/Address:			Analysis Required														
TECH TO STORY	Drive	)	198	-34	6			1						1	<u> </u>		
TRC 21 Technology 7 Truine, CA 9 Project Manager:	)	~ ~	<u> </u>	cke				terehlorate									ار ما ما ا
Truine, CH 9	1261	8		Number:	<u>ea</u>		[	3					1				H/T UP 3/24
Project Manager:	1		120		122/		×						1	ļ	ļ		
Richard Scot	7+				7 - 6	1336	١.	깈			}		1		}	ł	}
Sampler:			Fax Nu	mber;				5							ļ	•	
Sample	Sample	Container	# of	Samp	olina	Preservatives	1	2				}		}			
Description	Matrix		Cont.	Date/	-		(	3									Special Instructions
PS-15 / JB03526	Soil	* Gluss	1	2 24	199		5	(									* extract
PS-16 / J803527	,	1	\	1-1-	1		\										
PS-17 /1803528					7												
PS-18 /IB03529																	
PS-19/IB03530					1						~				1		
PS-20/180353/									:								
PS-21/JB03532																	
PS-22/ IB03533																	
PS-23 / JB03534																	
PS-24/IB03535																	
PS-25 /IB03536										!							
PS-26/IB03537																	
PS-27/IB03538																	
PS-28/IB03539-	4	V	7	8			7	/									
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						Ranka	גונ	u				160	9 <del>0</del> xw2	48 hours normal			<del></del>
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					i	mun	Received in Lab by Date /Time:  Fund Ruhman 3/12/99 1600						intact on ice				

## **Space Propulsion Operations**

P.O. Box 49028 San Jose, CA 95161-9028 408-779-9121



Mr. G. Nicoll United Technologies Corporation P.O. Box 49028 San Jose, CA 95161-9028

The following samples were received by the Chemical Systems Division Environmental Quality Laboratory (EQL) for analysis under California Department of Health Services (DHS) Environmental Laboratory Accreditation Program (ELAP) certificate number 1639:

COC: DMA14711

DATE SAMPLED: 02/24/99 PROJECT NO.: ESAR000796

LAB SAMPLE ID	ANALYSIS	LAB SAMPLE ID	ANALYSIS
BLANK	CLO4 (IC)	IB03530	CLO4 (IC)
IB03512	CLO4 (IC)	IB03531	CLO4 (IC)
IB03513	CLO4 (IC)	IB03532	CLO4 (IC)
IB03514	CLO4 (IC)	IB03533	CLO4 (IC)
IB03515	CLO4 (IC)	IB03534	CLO4 (IC)
IB03516	CLO4 (IC)	IB03535	CLO4 (IC)
IB03517	CLO4 (IC)	IB03536	CLO4 (IC)
IB03518	CLO4 (IC)	IB03537	CLO4 (IC)
IB03519	CLO4 (IC)	IB03538	CLO4 (IC)
IB03520	CLO4 (IC)	IB03539	CLO4 (IC)
IB03521	CLO4 (IC)	IB03551	CLO4 (IC)
IB03522	CLO4 (IC)	IB03552	CLO4 (IC)
IB03523	CLO4 (IC)	IB03553	CLO4 (IC)
IB03524	CLO4 (IC)	IB03554	CLO4 (IC)
IB03525	CLO4 (IC)	IB03555	CLO4 (IC)
IB03526	CLO4 (IC)	IB03793	CLO4 (IC)
IB03527	CLO4 (IC)	IB05CLO4C	CLO4 (IC)
IB03528	CLO4 (IC)	ICO6CLO4C	CLO4 (IC)
IB03529	CLO4 (IC)		

This report has been reviewed for technical correctness and completeness as verified by the signatures below. An attached QA/QC Report has a discussion of any analytical problems associated with these samples.

Any questions regarding this report should be directed to the Laboratory Director.

Bruce Boman, Chemist

**Environmental Quality Laboratory** 

Steven R. Secara, Laboratory Director

**Environmental Quality Laboratory** 

Date

# APPENDIX A.3 UNITED TECHNOLOGIES CORPORATION

American Pacific Corporation - Utah Operations 10622 West 6400 North, Cedar City, Utah 84720 Tel: (435) 865-5000 Fax: (435) 865-5029

Laboratory				. Р	roject No.: _	<u>98-346 l</u>	_ockheed	
(if not AMP	AC)			Re	port Date:	March :	22, 1999	
Client:		TRC		. ;	Sample ID:	PS-01/IB03512		
		21 Technology Drive Irvine, CA 92618		Re	ceipt Date:	March 12, 1999		
Collection	Date:	February 24, 1	999	Collec	NA			
Preservati	on:	Refrigeration	<u>1</u>	Prepara	ation Date:	March 16, 1999		
		1	est Resul	ts				
Analyte	Date	Method	MDL*	Units	Result	Analyst	Deviations	
Perchlorate	3/19/99	WEC-LAB-060-WI-54	1000	ppb	468,900	1350	NA	
Descriptio	n of Devi	ations and/or QC Failu	res:					
Authorize	d Signatu	re and Title:	Lent	l'u Ru	Vinsin			
			Director	· Analytical	Labs and Pr	oduct Dev	elopment	

## QA/QC REPORT **ENVIRONMENTAL QUALITY LABORATORY** (408) 776-4214

United Technologies P. O. Box 49028

WOR/C of C

: DMA14711

San Jose, CA 95161-9028

Date Sampled : ESAR000796

Project

: 02/24/99

## **QA/QC SUMMARY**

No QA/QC problems were associated with these samples.

All samples were analyzed within holding times.

# UNITED TECHNOLOGIES/CHEMICAL SYSTEMS DIVISION ENVIRONMENTAL QUALITY LABORATORY REPORT DESCRIPTION

All Official Analytical Reports (OARs) report amounts as gross values, i.e., <u>not</u> corrected for blank or surrogate recovery. Concentration values of tentatively identified compounds (TICs) reported as a result of the Library Search Compound Report (8260 analysis only) are approximate values only.

### SAMPLE TYPE

OARs may contain a comment regarding the type of sample analyzed for that report. A list of these abbreviations follows.

- S: Sample
- R: Replicate of Sample
- M: Matrix spike of listed sample
- B: System blank
- C: Continuing calibration standard
- L: Laboratory control standard
- E: External reference standard
- V: GC/MS verification analysis of Bromofluorobenzene (BFB)

### QUALIFIERS

OARs may contain abbreviations referring to compound concentrations or various qualifying statements concerning the compound concentrations reported. A list of these abbreviations follows.

- N/D or <: Compound was not detected at or above the listed reporting limit or concentration.
- U: Compound was not detected at or above listed reporting limit or concentration.
- B: Compound was found in associated system, trip, equipment, or field blank.
- E: Estimated. Reported concentration is outside the instrument's calibration range.
- J: Compound was detected below the listed reporting limit. Concentration should be treated as an estimated value. Tentatively Identified Compounds (TICs) will always have a "J" qualifier since they are not included in the instrument calibration.
- N: Indicates presumptive evidence of a compound. This flag is used only for TICs.

Absence of a qualifier indicates that the compound was detected at or above the listed reporting limit.

### SURROGATE RECOVERY SUMMARY AND MATRIX SPIKE RECOVERY FORMS

These pages contain Quality Assurance data. All values outside of established control limits will be flagged with an "\*". The total number of matrix spikes or relative percent differences outside of the limits will be listed at the bottom of the matrix spike recovery form.

## PERCHLORATE ANALYSIS DATA SHEET CSD WI 60.12.5W216.2 (EPA METHOD 300.0 modified) **ENVIRONMENTAL QUALITY LABORATORY** (408) 776-4214 **DHS-ELAP CERTIFICATE #1639**

PROJECT ID:ESAR000796

C of C#: DMA14711 DATE REPORT:03/25/99

MATRIX: WATER

Sample ID	Dilution	Detection	Reported	Reporting	Qualifier	Date	Date	Time
	factor	limit	concentration	units		Sampled	Analyzed	Analyzed
IB03512	50	200	515	mg/L			03/22/99	13:26
IB03513	50	200	236	mg/L			03/22/99	13:41
IB03514	50	200	773	mg/L			03/22/99	13:56
IB03515	50	200	630	mg/L			03/22/99	14:11
IB03515R	50	200	1184	mg/L			03/22/99	15:57
IB03516	50	200	476	mg/L			03/22/99	14:26
IB03517	1	4	368	mg/L			03/23/99	15:04
IB03518	50	200	883	mg/L			03/22/99	14:56
IB03519	1	4	96.8	mg/L			03/23/99	15:19
IB03520	50	200	512	mg/L			03/22/99	15:27
IB03521	1	4	<4	mg/L	U		03/23/99	15:35
IB03522	50	200	626	mg/L			03/22/99	16:42
IB03523	1	4	59.7	mg/L			03/23/99	15:50
IB03524	1	4	<4	mg/L	U		03/23/99	16:05
IB03525	50	200	864	mg/L			03/22/99	17:27
IB03526	50	200	519	mg/L			03/22/99	17:42
IB03527	50	200	564	mg/L			03/22/99	17:58
IB03528	1	4	<4	mg/L	U		03/23/99	16:20
IB03529	1	4	83.2	mg/L			03/24/99	10:56
IB03529R	1	4	99.3	mg/L			03/24/99	11:12
IB03530	50	200	327	mg/L			03/22/99	18:43
IB03531	50	200	1000	mg/L			03/22/99	
IB03532	50	200	256	mg/L			03/22/99	
IB03533	1	4	264.6	mg/L			03/24/99	11:42
IB03534	1	4	73.8	mg/L			03/24/99	
IB03535	10	40	284	mg/L			03/24/99	14:02
IB03536	50	200	368	mg/L			03/22/99	21:44
IB03537	50	200	481	mg/L			03/22/99	21:59
IB03538	10	40	712	mg/L			03/24/99	
IB03539	1	4	362.3	mg/L			03/24/99	
IB03551	50	200	529	mg/L			03/22/99	22:44
1B03552	50	200	2009	mg/L			03/22/99	
IB03553	10	40	1438	mg/L			03/23/99	14:04
IB03554	10	40	630	mg/L			03/23/99	14:19
IB03555	500	2000	2600	mg/L		1	03/24/99	
IB03793	10	40	3298	mg/L	1		03/23/99	

Reporting Limit for Perchlorate is 4 mg/L

## EQL QUALITY CONTROL DATA

Lac advicti out		Spike	Spike			
		Concentration	Recovery	!	Date	Time
Sample ID	(mg/L)	(mg/L)		QC Limits	Analyzed	Analyzed
300MB990318-1		<4		<4	03/18/99	13:49
300MB990322-1		<4		<4	03/22/99	20:28
300MB990322-1		<4		<4	03/22/99	11:44
300MB990323-1		<4		<4	03/24/99	10:41
300MLCS990318-1	9.95	10.2	103%	90% - 110%	03/18/99	18:36
300MLCS990318-2	9.95	10.6	107%	90% - 110%	03/18/99	22:40
300MLCS990322-1	9.95	9.56	96%	90% - 110%	03/22/99	19:58
300MLCS990322-2	9.95	9.44	95%	90% - 110%	03/24/99	10:11
300MLCS990323-1	48.35	53.2	110%	90% - 110%	03/24/99	12:57
300MLFB990318-1	19.6	19.2	98%	90% - 110%	03/18/99	18:25
300MLFB990318-2	19.6	18.1	92%	90% - 110%	03/18/99	22:29
300MLFB990322-1	19.6	17.7	90%	90% - 110%	03/22/99	19:43
300MLFB990322-2	38.4	38	99%	90% - 110%	03/24/99	09:56
300MLFB990323-1	38.4	34.5	90%	90% - 110%	03/24/99	14:32
300MLPC990318-1	100	109	109%	90% - 110%	03/18/99	16:07
300MLPC990318-2	100	108.6	109%	90% - 110%	03/18/99	18:46
300MLPC990318-3	100	108.3	108%	90% - 110%	03/18/99	21:15
300MLPC990322-1	100	98	98%	90% - 110%	03/22/99	11:29
300MLPC990322-2	100	102	102%	90% - 110%	03/22/99	16:27
300MLPC990322-3	100	105.2	105%	90% - 110%	03/22/99	20:13
300MLPC990322-4	100	103.4	103%	90% - 110%	03/23/99	13:49
300MLPC990322-5	100	99.4	99%	90% - 110%	03/24/99	08:56
300MLPC990323-1	100	103.9	104%	90% - 110%	03/24/99	10:26

## ACCOMPANYING QC SAMPLE DATA

Sample ID			% recovery		Date Analyzed	TIME
BLANK	0	0			03/15/99	16:37
IB05CLO4C	100	98.7	99%	90% - 110%	03/15/99	16:49
IB06CLO4C	100	94.3	94%	90% - 110%	03/15/99	17:02

### MATRIX SPIKE DATA

	Spike	Sample	Matrix Spike	Matrix			
	Added	Concen.	Concentration	Spike		Date	Time
Sample ID	(ug/L)	(ug/L)	(ug/L)	Recovery	QC Limits	Analyzed	Analyzed
IB03515MS	19600	630	18500	91%	75%-125%	03/22/99	16:12
IB03515MS	19600	630	19090	94%	75%-125%	03/18/99	15:56
IB03524MS	19600	0	17890	91%	75%-125%	03/22/99	19:28
IB03524MS	19600	0	18950	97%	75%-125%	03/18/99	18:14
IB03535MS	19600	284	19973	100%	75%-125%	03/22/99	23:30
IB03535MS	19600	284	18490	93%	75%-125%	03/18/99	21:04
IB03555MS	19200	2600	18320	82%	75%-125%	03/24/99	09:41
IB03555MS	19600	0	18940	97%	75%-125%	03/18/99	22:18

300MLPC...: Laboratory Performance Check sample performed to verify that the calibration curve is still valid.
300MLFB...: Laboratory Fortified Blank sample performed to verify recovery of analyte as it is taken through the system.
300MLCS...: Laboratory Control Sample performed to verify system operation. Sample is prepared using a stock standard other than the standard used to prepare the calibration curve.
...MS or ... MSD: Analytical samples spiked with analyte to determine matrix effect on analysis.

	Dol	$\Lambda / I$	Analy	tical	
		1 4 (	n idiy	ucui	•
V	2852 /	Iton Ave., i	rvine, CA 22508	(949) 261-1022	FAX (949) 261-12
40440	A	PU. 5 C		Anna) 476 4867	EAY (800) 479 48.

To: United Innologies Chemical Systems

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1014 E. COORY Dr., SUIR A. COROT, CA 82324 (197) 370-4607
10521 Sherman Way, Suire C-11, Van Nuye, CA 81406 (818) 771-1843
2465 W 12th St., Suire 1, Tempe, AZ 85281 (802) 868-8272
484 Cheeapeake Dr., Suire 805, San Diego, CA 82123 (818) 805-9586
FAX (818) 505-8688
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Page 1 of 3

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PS-03/1803514																
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Note: By relinquishing samples to Del Mar Analytical, client agrees to pay for the services requested on this chain of custody form and any additional analyses performed on this project. Payment for services is due within 30 days from the date of invoice. Sample(s) will be disposed of after 30 days.

FAX (949) 281-1228 1014 E. Cooley Dr., Suite A. Colton, CA 92324 (909) 370-4867 FAX (909) 370-1046 16525 Sherman Way, Suite C-11, Van Nuys, CA 91406 (818) 779-1844 FAX (818) 779-1845

2485 W 12th St., Suite 1, Tempe, AZ 85281 (602) 968-8272 FAX (602) 968-1338

## CHAIN OF CUSTODY FORM

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Note: By relinquishing samples to Del Mar Analytical, client agrees to pay for the services requested on this chain of custody form and any additional analyses performed on this project. Payment for services is due within 30 days from the date of invoice. Sample(s) will be disposed of after 30 days.

1014 E. Cooley Dr., Sulte A, Colton, CA 92324 (909) 370-4667 FAX (909) 370-1048 16525 Sherman Way, Suite C-11, Van Huys, CA 91406 (818) 779-1844 FAX (818) 779-1843

## CHAIN OF CHETODY FORM

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Note: By relinquishing samples to Del Mar Analytical, client agrees to pay for the services requested on this chain of custody form and any additional analyses performed on this project. Payment for services is due within 30 days from the date of invoice. Sample(s) will be disposed of after 30 days.

# APPENDIX A.4 MONTGOMERY WATSON LABORATORIES



a Division of Montgomery Watson Americas, Inc. 555 East Walnut Street Pasadena, California 91101 Te1: 626 568 6400 Fax: 626 568 6324 1 800 566 LABS (1 800 566 5227)

### Laboratory Report

for

AWWARF Perchlorate Project MW/PAS 2

Pasadena , CA 91101

Attention: Andy Eaton Fax: 626-568-6324

ADE Andy Eaton

Report#: 52565

CLO4



Laboratory Report #52565

a Division of Montgomery Watson Americas, Inc. 555 East Walnut Street Pasadena, California 91101 Te1: 626 568 6400 Fax: 626 568 6324 1 800 566 LABS (1 800 566 5227)

AWWARF Perchlorate Project Andy Eaton MW/PAS 2 Pasadena , CA 91101 Samples Received 12-mar-1999 13:37:03

Prepared Analyzed	QC Batch# Method	Analyte	Result	Units	MDL	Dilution
PS-01/IB03512	EXTRACT (99031232	3) Sampled on	02/24/99			
03/18/99	93708 ( MOD/EPA 300	) Perchlorate	530000	ug/l	40000	10000
PS-02/IB03513	EXTRACT (99031232	(4) Sampled on	02/24/99			
03/18/99		) Perchlorate	310000	ug/l	16000	4000
PS-03/IB03514	EXTRACT (99031232	25) Sampled on	02/24/99			
03/18/99	93708 ( MOD/EPA 300	•	940000	ug/1	40000	10000
PS-04/IB03515	EXTRACT (99031232	26) Sampled on	02/24/99			
03/18/99	93708 ( MOD/EPA 300		800000	ug/1	40000	10000
-05/TB03516	EXTRACT (99031232	27) Sampled on	02/24/99			
03/18/99	93708 ( MOD/EPA 300	<del>-</del>	620000	ug/l	40000	10000
DG N.E. / TR. N.3.51.7	' ፑሂጥኮኤሮጥ (ዓርበ3133	28) Sampled on	02/24/99			
03/18/99		) Perchlorate	300000	ug/l	40000	10000
DC 07/TD02E10	, EAGUD WAS (20021-22)	29) Sampled on	02/24/99			
03/18/99	•	) Perchlorate	500000	ug/l	40000	10000
DG 00/TD07F1	) TYMD3.CM /0003103:	20)	00/04/00			
PS-US/IBU35IS	94668 (MOD/EPA 300	•	02/24/99	ug/l	4800	1200
		,		<b>2</b> .		
•	EXTRACT (9903123:	•	, ,			
03/18/99	93708 ( MOD/EPA 300	) Perchlorate	430000	ug/l	40000	10000

Laboratory Report #52565

a Division of Montgomery Watson Americas, Inc. 555 East Walnut Street Pasadene, Celifornia 91101 Te1: 626 568 6400 Fax: 626 568 6324 1 800 566 LABS (1 800 566 5227)

# AWWARF Perchlorate Project (continued)

Prepared Analyzed	QC Batch# Method	Analyte	Result	Units	MDL	Dilution
•	EXTRACT (99031233		• •			
04/07/99	94669 ( MOD/EPA 300 )	Perchlorate	ND	ug/l	40	10
PS-11/IB03522	EXTRACT (99031233	3) Sampled on	02/24/99			
03/18/99	93708 ( MOD/EPA 300	) Perchlorate	880000	ug/l	40000	10000
PS-12/IB03523	EXTRACT (99031233	4) Sampled on	02/24/99			
03/18/99	93707 ( NOD/EPA 300	) Perchlorate	53000	ug/l	8000	2000
PS-13/IB03524	EXTRACT (99031233	5) Sampled on	02/24/99			
04/07/99	94669 ( MOD/EPA 300	) Perchlorate	(87)	ug/l	20	5
PS-14/IB03525	EXTRACT (99031233	66) Sampled on	02/24/99			
03/18/99	93708 ( MOD/EPA 300	) Perchlorate	900000	ug/l	80000	20000
PS-15/IB03526	EXTRACT (99031233	(7) Sampled on	02/24/99			
03/18/99	93708 ( MOD/EPA 300	) Perchlorate	610000	ug/l	40000	10000
PS-16/IB03527	EXTRACT (99031233	Sampled on	02/24/99			
03/18/99	93708 ( NOD/EPA 200	) Perchlorate	740000	ug/l	40000	10000
PS-17/IB03528	EXTRACT (99031233	39) Sampled on	02/24/99			
04/07/99	94669 ( NOD/EPA 300	) Perchlorate	220	ug/l	20	5
PS-18/IB03529	EXTRACT (99031234	10) Sampled on	02/24/99			
03/18/99	93707 ( MOD/EPA 300	•	75000	ug/1	8000	2000
PS-19/IB03530	EXTRACT (99031234	11) Sampled on	02/24/99			
03/18/99	93708 ( MOD/EPA 300	•	270000	ug/l	80000	20000



Laboratory Report #52565

MONTGOMERY WATSON LABORATORIES a Division of Montgomery Watson Americas, Inc. 555 East Walnut Street Pasadena, California 91101 Te1: 626 568 6400 Fax: 626 568 6324 1 800 566 LABS (1 800 566 5227)

## AWWARF Perchlorate Project (continued)

Prepared	Analyzed	QC Batch#	Method	Analyte			Result	Units	MDL	Dilution
PS-20/I	B03531	EXTRACT	(9903	12342)	-	on	02/24/99	ug/l	40000	10000
			• • • • • • • • • • • • • • • • • • • •					α <b>g</b> /Ι	40000	10000
PS-21/I			•	12343)	-	on	02/24/99			
	03/18/99	93708	( MOD/EPA	300 ) Perchlors	ite		290000	ug/l	40000	10000
PS-22/I	B03533	EXTRACT	(9903	12344)	${\tt Sampled}$	on	02/24/99			
	03/18/99	93707	( MOD/EPA	300 ) Perchlora	ite		140000	ug/l	8000	2000
PS-23/I	B03534	EXTRACT	(9903	12345)	Sampled	on	02/24/99			
•	03/18/99	93707	( MOD/EPA	300 ) Perchlore	ate		34000	ug/1	8000	2000
PS-24/T	B03535	EXTRACT	' (9903	12346)	Sampled	on	02/24/99			
	03/18/99		•	300 ) Perchlore	-	<b></b>	220000	ug/1	16000	4000
20 25 /5	20050		. (0000	10245	<b>6 3</b> - <b>3</b>		00/04/00			
-	03/18/99	93708	-	12347) 300 ) Perchlors	-	on	02/24/99	ug/l	40000	10000
			•	·				-3/-	10000	2000
PS-26/I			•	12348)	Sampled	on	02/24/99			
	03/18/99	93708	( MOD/EPA	100 ) Perchlor	ate		320000	ug/l	40000	10000
PS-27/I	B03538	EXTRACI	(9903	12349)	Sampled	on	02/24/99			
	03/18/99	93708	( MOD/EPA	300 ) Perchlor	ate		550000	ug/l	40000	10000
PS-28/I	:B03539	EXTRACT	(9903	12350)	Sampled	on	02/24/99			
·	03/18/99	93708		300 ) Perchlor	_		220000	ug/l	40000	10000
PS-29/T	B03551	EXTRACI	ר (פפחז	12351)	Sampled	On	02/24/99			
	03/18/99	93708	( NOD/EPA	<del>-</del>	-	<b>011</b>	540000	ug/l	40000	10000



Laboratory Report #52565

a Division of Montgomery Watson Americas, Inc. 555 East Walnut Street Pasadena, California 91101 Te1: 626 568 6400 Fax: 626 568 6324 1 800 566 LABS (1 800 566 5227)

# AWWARF Perchlorate Project (continued)

Prepared Analyzed	QC Batch# Method	Analyte	Result	Units	MDL	Dilution
PS-30/IB03552	EXTRACT (99031235	2) Sampled on	02/24/99	ug/1	160000	40000
PS-31/IB03553	EXTRACT (99031235	3) Sampled on Perchlorate	02/24/99	ug/l	160000	40000
PS-32/IB03554	EXTRACT (99031235	4) Sampled on Perchlorate	02/24/99	ug/1	40000	10000
PS-33/IB03555	EXTRACT (99031235	5) Sampled on Perchlorate	02/24/99	ug/l	40000	10000
PS-34/IB03793	EXTRACT (99031235	6) Sampled on Perchlorate	02/18/99	ug/l	800000	200000
BLANK (990312 03/18/99	357) Sampled o	·	, ND	ug/l	4.0	1
LCS1 100 PPB		impled on 02/24/99 Perchlorate	100	ug/1	8.0	2
LCS2 100 PPB 03/18/99		empled on 02/24/99 Perchlorate	99	ug/l	8.0	2



Report Comments #52565

a Division of Montgomery Watson Americas, Inc. 555 East Walnut Street Pasadena, California 91101 Tel: 626 568 6400 Fax: 626 568 6324 1 800 566 LABS (1 800 566 5227)

(990312332)

CLO4

This sample was pre-treated prior to the analysis.

(990312335)

CLO4

This sample was pre-treated prior to the analysis. The reported result is reported as an estimate due to reclining base line. If diluted or pre-treated more sample would possibly come out with ND with a higher reporting limit.

(990312339)

CLO4

This sample was pretreated prior to the analysis.

Laboratory QC Report #52565

a Division of Montgomery Watson Americas, Inc. 555 East Walnut Street Pasadena, California 91101 Te1: 626 568 6400 Fax: 626 568 6324 1 800 566 LABS (1 800 566 5227)

## AWWARF Perchlorate Project

	QC Batch #93707	Perchlorate		
QC	Analyte		ield (%) Limits (%)	RPD (%)
LCS1	Perchlorate	20.0 19.5 97.5	•	
LCS2	Perchlorate	20.0 21.7 108.	5 (90.00 - 110.00	0 ) 11
MBLK	Perchlorate	ND		
	QC Batch #93708	Perchlorate		
δc	Analyte	Spiked Recovered N	field (%) Limits (%)	RPD (%)
LCS1	Perchlorate	20.0 19 95.0	90.00 - 110.0	0 )
LCS2	Perchlorate	20.0 19.6 98.0	( 90.00 - 110.0	0 ) 3.1
MBLK	Perchlorate	ND		
	QC Batch #94668	Perchlorate		
QC	Analyte	Spiked Recovered	(ield (%) Limits (%)	RPD (%)
LCS1	Perchlorate	20.0 19.8 99.0	( 90.00 - 110.0	0 )
LCS2	Perchlorate	20.0 20.1 100	.5 ( 90.00 - 110.0	0 ) 1.5
MBLK	Perchlorate	ND		
	QC Batch #94669	Perchlorate		
QC	Analyte	Spiked Recovered	Yield (%) Limits (%)	RPD (%)
rcsi	Perchlorate	50.0 46.2 92.	4 (90.00 - 110.0	0 }
LCS2	Perchlorate	100 102 102	.0 ( 90.00 - 110.0	0)
MBLK	Perchlorate	ND		

Spikes which exceed Limits and Method Blanks with positive results are highlighted by <u>Underlining.</u>
Criteria for MS and DUP are advisory only and not applicable for ICR monitoring.

APPENDIX A.5
THIOKOL CORPORATION

## CERTIFICATE OF ANALYSIS

Listing of sample Information and Testing Requested

Monday, March 29, 1999

TESTED FOR Thiokol: DLV

**Environmental Monitoring** 

M/S 301

Thiokol Corp, UT 84322

Paul Hancock

Project: Misc. Environmental Testing

ANALYZED BY Thiokol Environmental Laboratory

Thiokol Corporation

P.O. Box 707 M/S 245

Brigham City, UT 84302-0707

801-863-3732

800-863-8080

Login Number L2200

Lab No Client Number Test Requested	Description	Received CollectDate/Time Matrix
L2200-1 PS-01/ IBO3512		03/15/99 02/24/99 00:00
SOP 302	Perchiorate - Ion Chromatography	Water
L2200-2 PS-02/ IBO3513		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-3 PS-03/ IBO3514		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-4 PS-04/ IBO3515		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-5 PS-05/ IBO3516	<u> </u>	03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-6 PS-06/ IBO3517		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-7 PS-07/ IBO3518		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-8 PS-08/ IBO3519		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-9 PS-09/ IBO3520		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-10 PS-10/ IBO3521		03/15/99 02/24/99 00:00
SOP 361	Perchlorate - Ion Chromatography	Water
L2200-11 PS-11/ IBO3522		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-12 PS-12/ IBO3523		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-13 PS-13/ IBO3524		03/15/99 02/24/99 00:00
SOP 361	Perchiorate - Ion Chromatography	Water
L2200-14 PS-14/ IBO3525		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-15 PS-15/ IBO3526		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-16 PS-16/ IBO3527		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-17 PS-17/ IBO3528		03/15/99 02/24/99 00:00
SOP 361	Perchlorate - Ion Chromatography	Water
L2200-18 PS-18/ IBO3529		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-19 PS-19/ IBO3530		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-20 PS-20/ IBO3531		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-21 PS-21/ IBO3532		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - ion Chromatography ,	Water

L2200-22 PS-22/ IBO3533		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-23 PS-23/ IBO3534		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-24 PS-24/ IBO3535		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-25 PS-25/ IBO3536		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-26 PS-26/ IBO3537		03/15/99 02/24/99 00:00
SOP 302	Perchiorate - Ion Chromatography	Water
L2200-27 PS-27/ IBO3538		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-28 PS-28/ IBO3539		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-29 PS-29/ IBO3551		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-30 PS-30/ IBO3552		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-31 PS-31/IBO3553		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-32 PS-32/ IBO3554		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-33 PS-33/ IBO3555		03/15/99 02/24/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-34 PS-34/ IBO3793		03/15/99 02/18/99 00:00
SOP 302	Perchlorate - Ion Chromatography	Water
L2200-35 BLANK		03/15/99
SOP 361	Perchlorate - Ion Chromatography	Water
L2200-36 LCS1 100PPB		03/15/99
SOP 361	Perchlorate - Ion Chromatography	Water
L2200-37 LCS2 100PPB		03/15/99
SOP 361	Perchlorate - Ion Chromatography	Water

### Comments:

PS-10, PS-13, PS-17, PS-35, PS-36, and PS-37: These samples were analyzed using a more sensitive method. Their results ere reported in Micrograms/Liter rather than Milligrams/Liter. PS-10, PS-13, and PS-17: These samples were diluted to remove interfering matrix effects. PS-36 and PS-37: These samples were diluted to bring their value within the calibration range of the method used.

Certified By James M. Anderson, QC Officer

This certifies that the following samples were analyzed using good laboratory practices to show the following results:

## Listing of Results by Sample

- I ID. DO 04/1000540				-6-10-1	0000 4		Callant Dates Of	2/24/00 00:00
Sample ID: PS-01/ IBO3512			Li	ab ID: L		`	Collect Date: 02	2/24/99 00:00
Test Parameter	Result	Units	MDL	EQL	Dil. Factor	Method	Analyst	Test Date
PERCHLORATE	471	mg/l	15	75	100	SOP 302	cws	03/19/99 16:01
Sample ID: PS-02/ IBO3513			1	ab ID: L	2200-2		Collect Date: 02	2/24/99 00:00
Sample ID. 113-02 IBOSS13			<u> </u>	ab 10. L	Dil.		Ooneer Date. O	224/03/00:00
Test Parameter	Result	Units	MDL	EQL	Factor	Method	Analyst	Test Date
PERCHLORATE	324	mg/i	15	75	100	SOP 302	cws	03/19/99 16:15
Sample ID: PS-03/ IBO3514			L	ab ID: L	2200-3		Collect Date: 0	2/24/99 00:00
		<del></del>		-	Dil.			
<u>Test Parameter</u>	Result	<u>Units</u>	MDL	<u>EQL</u>	Factor	<u>Method</u>	<u>Analyst</u>	Test Date
PERCHLORATE	886	mg/l	15	75	100	SOP 302	cws	03/19/99 16:29
Sample ID: PS-04/ IBO3515			L	ab ID: l	.2200-4		Collect Date: 0	2/24/99 00:00
	1				Dil.	· · · · · · · · · · · · · · · · · · ·		
<u>Test Parameter</u>	Result	<u>Units</u>	MDL	<u>EQL</u>	<u>Factor</u>	Method	<u>Analys</u>	t Test Date
PERCHLORATE	766	mg/l	15	75	100	SOP 302	cws	03/19/99 16:43
Sample ID: PS-05/ IBO3516			L	ab ID: I	2200-5		Collect Date: 0	2/24/99 00:00
					Dil.			
<u>Test Parameter</u>	Result	<u>Units</u>	MDL	<u>EQL</u>	Factor	Method	<u>Analys</u>	
PERCHLORATE	636	mg/l	15	75	100	SOP 302	cws	03/19/99 16:57
Sample ID: PS-06/ IBO3517				ab ID:	L2200-6		Collect Date: 0	2/24/99 00:00
Total December	D		MOI	<b>50</b> 1	Dil.	34 - 41	A t	4 Tark Data
<u>Test Parameter</u> PERCHLORATE	Result 309	<u>Units</u>	<u>MDL</u> 15	<u>EQL</u> 75	Factor 100	Method SOP 302	<u>Analys</u> CWS	
PERCHLORATE	309	mg/l	15	15	100	30F 30Z	CVVS	03/19/99 17:11
Sample ID: PS-07/ IBO3518	-		1	_ab ID:	L2200-7		Collect Date: 0	2/24/99 00:00
	Recult	Unite			Dil.	Method		
Sample ID: PS-07/ IBO3518  Test Parameter PERCHLORATE	Result 492	<u>Units</u>	MDL	_ab ID: <u>EQL</u> 75	Dil. Factor	Method SOP 302		t <u>Test Date</u>
Test Parameter PERCHLORATE		<u>Units</u> mg/l	<u>MDL</u> 15	<u>EQL</u> 75	Dil. <u>Factor</u> 100		<u>Analys</u> CWS	t <u>Test Date</u> 03/19/99 17:25
Test Parameter			<u>MDL</u> 15	<u>EQL</u> 75	Dil. Factor 100 L2200-8		Analys	t <u>Test Date</u> 03/19/99 17:25
<u>Test Parameter</u> PERCHLORATE			<u>MDL</u> 15	<u>EQL</u> 75	Dil. <u>Factor</u> 100		Analys CWS Collect Date: (	<u>Test Date</u> 03/19/99 17:25 02/24/99 00:00
Test Parameter PERCHLORATE Sample ID: PS-08/ IBO3519	492	mg/l	<u>MDL</u> 15	EQL 75 Lab ID:	Dil. Factor 100 L2200-8 Dil. Factor	SOP 302	Analys CWS Collect Date: (	t <u>Test Date</u> 03/19/99 17:25 02/24/99 00:00 tt <u>Test Date</u>
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE	492 Result	mg/I	MDL 15 MDL 3	<u>EQL</u> 75 Lab ID: <u>EQL</u> 15	Dil. Factor 100  L2200-8  Dil. Factor 20	SOP 302	Analys CWS Collect Date: ( Analys CWS	1 Test Date 03/19/99 17:25 02/24/99 00:00 11 Test Date 03/19/99 18:21
Test Parameter PERCHLORATE Sample ID: PS-08/ IBO3519 Test Parameter	492 Result	mg/I	MDL 15 MDL 3	<u>EQL</u> 75 Lab ID: <u>EQL</u> 15	Dil. Factor 100 L2200-8 Dil. Factor	SOP 302	Analys CWS Collect Date: (	1 Test Date 03/19/99 17:25 02/24/99 00:00 11 Test Date 03/19/99 18:21
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE	492 Result	mg/I	MDL 15 MDL 3	<u>EQL</u> 75 Lab ID: <u>EQL</u> 15	Dil. Factor 100 L2200-8 Dil. Factor 20 L2200-9	Method SOP 302	Analys CWS Collect Date: ( Analys CWS Collect Date: (	1 Test Date 03/19/99 17:25 02/24/99 00:00 11 Test Date 03/19/99 18:21 02/24/99 00:00
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE  Sample ID: PS-09/ IBO3520	492  Result 101	mg/l <u>Units</u> mg/l	MDL 15 I MDL 3	EQL 75 Lab ID: EQL 15 Lab ID:	Dil. Factor 100 L2200-8 Dil. Factor 20 L2200-9 Dil. Factor	Method SOP 302	Analys CWS Collect Date: ( Analys CWS Collect Date: (	t Test Date 03/19/99 17:25 02/24/99 00:00 t Test Date 03/19/99 18:21 02/24/99 00:00
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE  Sample ID: PS-09/ IBO3520  Test Parameter	Result 101	Units mg/l	MDL 15 MDL 3 MDL 15	EQL 75 Lab ID: 15 Lab ID: EQL 75	Dil. Factor 100 L2200-8 Dil. Factor 20 L2200-9 Dil. Factor	Method SOP 302	Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys	t Test Date 03/19/99 17:25 02/24/99 00:00 tt Test Date 03/19/99 18:21 02/24/99 00:00 tt Test Date 03/19/99 18:35
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE  Sample ID: PS-09/ IBO3520  Test Parameter PERCHLORATE  Sample ID: PS-10/ IBO3521	Result 101 Result 438	Units mg/l	MDL 15 MDL 3 MDL 15	EQL 75 Lab ID: 15 Lab ID: EQL 75	Dil. Factor 100 L2200-8 Dil. Factor 20 L2200-9 Dil. Factor 100	Method SOP 302 Method SOP 302	Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: (	t Test Date 03/19/99 17:25 02/24/99 00:00 t Test Date 03/19/99 18:21 02/24/99 00:00 t Test Date 03/19/99 18:35
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE  Sample ID: PS-09/ IBO3520  Test Parameter PERCHLORATE  Sample ID: PS-10/ IBO3521  Test Parameter	Result 101  Result 438  Result	Units mg/l	MDL 3 MDL 15 MDL 15	EQL 75 Lab ID: 15 Lab ID: EQL 75 Lab ID:	Dil. Factor 100 L2200-8 Dil. Factor 20 L2200-9 Dil. Factor 100 L2200-10 Dil. Factor	Method SOP 302 Method SOP 302	Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys	1 Test Date 03/19/99 17:25 02/24/99 00:00 11 Test Date 03/19/99 18:21 02/24/99 00:00 11 Test Date 03/19/99 18:35 02/24/99 00:00
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE  Sample ID: PS-09/ IBO3520  Test Parameter PERCHLORATE  Sample ID: PS-10/ IBO3521	Result 101 Result 438	Units mg/l	MDL 15  MDL 3  MDL 15	EQL 75 Lab ID: 15 Lab ID: EQL 75 Lab ID:	Dil. Factor 100 L2200-8 Dil. Factor 20 L2200-9 Dil. Factor 100 L2200-10 Dil. Factor	Method SOP 302 Method SOP 302	Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: (	1 Test Date 03/19/99 17:25 02/24/99 00:00 11 Test Date 03/19/99 18:21 02/24/99 00:00 11 Test Date 03/19/99 18:35 02/24/99 00:00
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE  Sample ID: PS-09/ IBO3520  Test Parameter PERCHLORATE  Sample ID: PS-10/ IBO3521  Test Parameter	Result 101  Result 438  Result	Units mg/l  Units mg/l	MDL 3 MDL 15 MDL 15 MDL 300	EQL 75 Lab ID: 15 Lab ID: EQL 75 Lab ID: EQL 1000	Dil. Factor 100 L2200-8 Dil. Factor 20 L2200-9 Dil. Factor 100 L2200-10 Dil. Factor	Method SOP 302 Method SOP 302	Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys	t Test Date 03/19/99 17:25 02/24/99 00:00 t Test Date 03/19/99 18:21 02/24/99 00:00 t Test Date 03/19/99 18:35 02/24/99 00:00
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE  Sample ID: PS-09/ IBO3520  Test Parameter PERCHLORATE  Sample ID: PS-10/ IBO3521  Test Parameter PERCHLORATE  Sample ID: PS-11/ IBO3522	Result 101  Result 438  Result U	Units mg/l  Units mg/l  Units ug/l	MDL 3 MDL 15 MDL 15 MDL 300	EQL 75 Lab ID: 15 Lab ID: 15 Lab ID: 1000 Lab ID:	Dil. Factor 20 L2200-9 Dil. Factor 100 L2200-10 Dil. Factor 100 L2200-10 Dil. Factor 100 L2200-11 Dil. Factor 100	Method SOP 302 Method SOP 302 Method SOP 361	Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: (	1 Test Date 03/19/99 17:25 02/24/99 00:00 11 Test Date 03/19/99 18:21 02/24/99 00:00 12 Test Date 03/19/99 18:35 02/24/99 00:00 13 Test Date 03/26/99 14:23
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE  Sample ID: PS-09/ IBO3520  Test Parameter PERCHLORATE  Sample ID: PS-10/ IBO3521  Test Parameter PERCHLORATE  Sample ID: PS-11/ IBO3522  Test Parameter	Result 101  Result 438  Result U	Units mg/l  Units mg/l  Units ug/l	MDL 3 MDL 15 MDL 300 MDL	EQL 15 Lab ID: EQL 1000 Lab ID: EQL 1000 Lab ID:	Dil. Factor 20 L2200-9 Dil. Factor 100 L2200-10 Dil. Factor 100 L2200-10 Dil. Factor 100 L2200-11 Dil. Factor 100	Method SOP 302  Method SOP 302  Method SOP 361	Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS	t Test Date 03/19/99 17:25 02/24/99 00:00 t Test Date 03/19/99 18:21 02/24/99 00:00 t Test Date 03/19/99 18:35 02/24/99 00:00 t Test Date 03/26/99 14:23 02/24/99 00:00
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE  Sample ID: PS-09/ IBO3520  Test Parameter PERCHLORATE  Sample ID: PS-10/ IBO3521  Test Parameter PERCHLORATE  Sample ID: PS-11/ IBO3522	Result 101  Result 438  Result U	Units mg/l  Units mg/l  Units ug/l	MDL 3 MDL 15 MDL 15 MDL 300	EQL 15 Lab ID: EQL 1000 Lab ID: EQL 1000 Lab ID:	Dil. Factor 20 L2200-9 Dil. Factor 100 L2200-10 Dil. Factor 100 L2200-10 Dil. Factor 100 L2200-11 Dil. Factor 100	Method SOP 302 Method SOP 302 Method SOP 361	Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: (	t Test Date 03/19/99 17:25 02/24/99 00:00 t Test Date 03/19/99 18:21 02/24/99 00:00 t Test Date 03/19/99 18:35 02/24/99 00:00 t Test Date 03/26/99 14:23 02/24/99 00:00
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE  Sample ID: PS-09/ IBO3520  Test Parameter PERCHLORATE  Sample ID: PS-10/ IBO3521  Test Parameter PERCHLORATE  Sample ID: PS-11/ IBO3522  Test Parameter	Result 101  Result 438  Result U	Units mg/l  Units mg/l  Units ug/l	MDL 3 MDL 15 MDL 300 MDL 315	EQL 75 Lab ID:  EQL 75 Lab ID:  EQL 1000 Lab ID:  EQL 75	Dil. Factor 20 L2200-9 Dil. Factor 100 L2200-10 Dil. Factor 100 L2200-10 Dil. Factor 100 L2200-11 Dil. Factor 100	Method SOP 302  Method SOP 302  Method SOP 361  Method SOP 302	Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS	t Test Date 03/19/99 17:25 02/24/99 00:00 t Test Date 03/19/99 18:21 02/24/99 00:00 t Test Date 03/19/99 18:35 02/24/99 00:00 t Test Date 03/26/99 14:23 02/24/99 00:00 t Test Date 03/26/99 19:03
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE  Sample ID: PS-09/ IBO3520  Test Parameter PERCHLORATE  Sample ID: PS-10/ IBO3521  Test Parameter PERCHLORATE  Sample ID: PS-11/ IBO3522  Test Parameter PERCHLORATE  Sample ID: PS-11/ IBO3522  Test Parameter PERCHLORATE  Sample ID: PS-12/ IBO3523	Result 101  Result 438  Result U  Result 529	Units mg/l  Units mg/l  Units ug/l	MDL 3 MDL 15 MDL 300 MDL 15	EQL 75 Lab ID: EQL 75 Lab ID: EQL 1000 Lab ID: EQL 75 Lab ID:	Dil. Factor 20 L2200-9 Dil. Factor 100 L2200-10 Dil. Factor 100 L2200-10 Dil. Factor 100 L2200-11 Dil. Factor 100 L2200-11 Dil. Factor 100 L2200-11 Dil. Factor 100 L2200-11	Method SOP 302  Method SOP 302  Method SOP 361  Method SOP 302	Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( COLLECT Date: ( CWS CWS COLLECT Date: ( CWS CWS CWS COLLECT Date: ( CWS CWS CWS CWS CWS CWS CWS CWS CWS CWS	1 Test Date 03/19/99 17:25 02/24/99 00:00 11 Test Date 03/19/99 18:21 02/24/99 00:00 12 Test Date 03/19/99 18:35 02/24/99 00:00 13 Test Date 03/26/99 14:23 02/24/99 00:00 14 Test Date 03/26/99 19:03 02/24/99 00:00
Test Parameter PERCHLORATE  Sample ID: PS-08/ IBO3519  Test Parameter PERCHLORATE  Sample ID: PS-09/ IBO3520  Test Parameter PERCHLORATE  Sample ID: PS-10/ IBO3521  Test Parameter PERCHLORATE  Sample ID: PS-11/ IBO3522  Test Parameter PERCHLORATE  Sample ID: PS-11/ IBO3522	Result 101  Result 438  Result U	Units mg/l  Units mg/l  Units ug/l	MDL 3 MDL 15 MDL 300 MDL 315	EQL 15 Lab ID: EQL 1000 Lab ID: EQL 75 Lab ID:	Dil. Factor 20 L2200-9 Dil. Factor 100 L2200-10 Dil. Factor 100 L2200-11 Dil. Factor 100 L2200-12 Dil. Factor 100 L2200-12 Dil. Factor 100 L2200-12 Dil. Factor 100 L2200-12 Dil. Factor 100	Method SOP 302  Method SOP 302  Method SOP 361  Method SOP 302	Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS Collect Date: ( Analys CWS	t Test Date 03/19/99 17:25 02/24/99 00:00 t Test Date 03/19/99 18:21 02/24/99 00:00 t Test Date 03/19/99 18:35 02/24/99 00:00 t Test Date 03/26/99 14:23 02/24/99 00:00 st Test Date 03/19/99 19:03 02/24/99 00:00

						<u></u>	<b></b>	70.4100.05.55
Sample ID: PS-13/ IBO3524			Li	ab ID: L	2200-13		Collect Date: 02	2/24/99 00:00
Test Parameter	Result	Units	MDL	EQL	Dil. Factor	Method	Analyst	Test Date
PERCHLORATE	962	ug/l	300	1000	100	SOP 361	cws	03/26/99 15:05
Sample ID: PS-14/ IBO3525		<u> </u>	1	ab ID· I	2200-14	· · · · · · · · · · · · · · · · · · ·	Collect Date: 0	2/24/99 00:00
Guilipie 10- 1 0-14/10/00/20					Dil.			
<u>Test Parameter</u>	Result	<u>Units</u>	MDL	EQL	Factor_	Method	Analyst	Test Date
PERCHLORATE	992	mg/l	15	75	100	SOP 302	cws	03/19/99 19:45
Sample ID: PS-15/ IBO3526			L	ab ID: L	2200-15		Collect Date: 0	2/24/99 00:00
					Dil.			
<u>Test Parameter</u>	Result	<u>Units</u>	MDL	<u>EQL</u>	Factor	Method	<u>Analys</u>	Test Date
PERCHLORATE	608	mg/i	15	75	100	SOP 302	cws	03/19/99 19:59
Sample ID: PS-16/ IBO3527			L	ab ID: L	2200-16		Collect Date: 0	2/24/99 00:00
					Dil.			
<u>Test Parameter</u>	Result	<u>Units</u>	MDL	<u>EQL</u>	Factor	<u>Method</u>	<u>Analys</u>	t Test Date
PERCHLORATE	721	mg/l	15	75	100	SOP 302	cws	03/19/99 20:13
Sample ID: PS-17/ IBO3528			L	ab ID: i	2200-17		Collect Date: 0	2/24/99 00:00
					Dil.			
Test Parameter	<u>Result</u>	<u>Units</u>	MDL	EQL	Factor	Method	Analys	
PERCHLORATE	U	ug/l	300	1000	100	SOP 361	cws	03/26/99 15:46
Sample ID: PS-18/ IBO3529				ab ID: I	L2200-18		Collect Date: C	2/24/99 00:00
To at Danamastan	Danulk	l Imita	Man	<b>50</b> 1	Dil.	Madhad	Amalua	4 Tool Date
Test Parameter PERCHLORATE	Result 69	<u>Units</u>	MDL 3	<u>EQL</u> 15	Factor 20	Method SOP 302	Analys CWS	t <u>Test Date</u> 03/19/99 21:22
	03	mg/l				307 302		
Sample ID: PS-19/ IBO3530			L	_ab ID:	L2200-19		Collect Date: (	02/24/99 00:00
Test Parameter	Result	Units	MDL	EQL	Dil. Factor	Method	Analys	t Test Date
PERCHLORATE	266	mg/l	15	<u> 75</u>		SOP 302	CWS	
Sample ID: PS-20/ IBO3531		<u> </u>			L2200-20		Collect Date: (	
				au IU.	Dil.		Concer Date.	22700 00,00
<u>Test Parameter</u>	Result	<u>Units</u>	MDL	<u>EQL</u>	Factor	Method	Analys	t Test Date
PERCHLORATE	556	mg/l	15	75		SOP 302	cws	03/19/99 21:50
Sample ID: PS-21/ IBO3532				Lab ID:	L2200-21		Collect Date:	02/24/99 00:00
					Dil.			
<b>Test Parameter</b>	Result	<u>Units</u>	MDL	EQL	Factor	Method	<u>Analys</u>	st Test Date
PERCHLORATE	300	mg/l	15	75	100	SOP 302	CWS	03/19/99 22:04
Sample ID: PS-22/ IBO3533				Lab ID:	L2200-22		Collect Date:	02/24/99 00:00
					Dil.			
Test Parameter	Result	<u>Units</u>	MDL	EQL	Factor			-,
PERCHLORATE	153	mg/l	3	15	20	SOP 302	CWS	03/19/99 22:18
Sample ID: PS-23/ IBO3534	·			Lab ID:	L2200-23	3	Collect Date:	02/24/99 00:00
Tast Baran - to-	D	11-24			Dil.	50-44		-4 Ta-4 D-4-
<u>Test Parameter</u> PERCHLORATE	Result 43	<u>Units</u>	MDL					st Test Date
	43	mg/l	3		5 20	SOP 302		
Sample ID: PS-24/ IBO3535				Lab ID:	L2200-24	\$	Collect Date:	02/24/99 00:00
Test Parameter	Doculé	l laite	, and	EOI	Dil.	Matha	d Analu	et Toet Date
PERCHLORATE	Result 248	<u>Units</u> mg/l	<u>MDL</u> 15		Factor 5 100	Method SOP 302		
	240	riig/i	13					
Sample ID: PS-25/ IBO3536				Lab iD:	L2200-2	5	Collect Date:	02/24/99 00:00
Test Parameter	Result	<u>Units</u>	MDL	EQL	Dil. Factor	Method	d Analy	st Test Date
PERCHLORATE	431	mg/i		. <u></u>		SOP 302		
	,	9.1			_ ,	402		

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3/20/99 12:24
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3/20/99 12:38
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3/20/99 01:00 4/99 00:00 Test Date 3/20/99 02:00 Test Date 3/20/99 02:1 8/99 00:00
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3/20/99 01:00 4/99 00:00  Test Date 3/20/99 02:00  Test Date 3/20/99 02:11 8/99 00:00  Test Date 3/20/99 02:3  Test Date
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## **Data Reporting Qualifiers**

- U Indicates compound was analyzed for and was not detected, or the compound was detected but was below the MDL.
- B Gas Chromatography and Mass Spectral Data Flag. This flag is used when an analyte is found in the blank as well as the sample
- J Gas Chromatography and Mass Spectral Data Flag. Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds or when the data indicated the presence of a compound that meets the identification criteria but the result is less than the EQL (e.g. 3 J with an EQL of 10)

MDL Method Detection Limit: The minimum concentration of a substance that can be confidently measured and reported. The laboratory has demonstrated that the MDL can be achieved in a laboratory reagent blank, but does not guarentee it can be achieved in all sample matrices. The MDL is approximately three times the signal noise level.

EQL Estimated Quantitation Limit: The EQL generally is 5 to 10 times the MDL. For many analytes the EQL is selected as the value of the lowest standard in the calibration curve.

Dil. Factor Dilution Factor: The prepared smple was diluted by this factor because the sample was too concentrated or due to other interferences in the sample matrix. Any dilution factor causes an appropriate increase in the MDL and EQL.

lar Analytical

1014 E. Cooley Dr., Suite A, Colton, CA 92324 (909) 370-4667 FAX (909) 370-1046 To: THOKOI Propulsion Group L220 14711.

CHAIN OF CUSTODY FORM Attn: Paul Hunicik Page 1 of 3 FAX (818) 779-1843 16525 Sherman Way, Sulte C-11, Van Nuye, CA 91406 (818) 779-1844 FAX (802) 968-1338 2465 W 12th St., Sulte 1, Tempe, AZ 85281 (602) 968-8272 FAX (819) 505-9089 9484 Chesapeake Dr., Sulte 805, Sen Diego, CA 82123 (819) 505-9596 Project/PO Number: Client Name/Address: Analysis Required TRC 98-346 Zi Technology Drive Irvine, CA 42618 Project Manager: H/TUP: 3/2. Loacheed
Phone Number: Richard Scott 949) 727-9336 Fax Number: Sample | Container # of Sampling Preservatives Sample Special Instructions Description Matrix Type Cont. Date/Time PS-01/1803512 Soil \* extract Gluss PS-02 /1803513 PS-03/1803514 PS-04/1803515 15-05 /1803516 PS-06/IB03517 PS07/78035/8 DS-08/1803519 15-09 /IB03520 75-10 / IB03521 17805522 PS-12/IB03523 PS-13 /IB03524 丁803525 Relinquished By: Date /Time: Received by: Turnaround Time: (Check) Received by Howicz Date /Time: 3/11/99

Received by Howicz Date /Time: 3/12/99 1460

History Howicz Date /Time: 3/12/99 1460 3/11/99 same day 72 hours \_\_\_\_ Relinquished By: 24 hours 5 days Relinquished By: Date /Time: Received in Lab by: Sample Integrity: (Check) Raul Hamasel Note: By relinquishing samples to Del Mar Analytical, client agrees to pay for the services requested on this chain of custody form and any additional analyses performed on this project. Payment for services is

due within 30 days from the date of invoice. Sample(s) will be disposed of after 30 days.



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Page Z of 3

2852 Alton Ave., Irvine, CA 92806 (949) 281-1022 FAX (949) 281-1228 1814 E. Cooley Dr., Suite A. Colton, CA 92324 (909) 378-4867 FAX (909) 378-1048 16525 Sherman Way, Suite C-11, Van Nuye, CA 91406 (618) 779-1844 FAX (618) 779-1843 2465 W 12th St., Suite 1, Tempe, AZ 85281 (602) 968-8272 FAX (602) 968-1338 8484 Cheseneaka Dr., Sulta 805, San Diago, CA 92123 (819) 505-9596 FAX (819) 505-9689

CHAIN OF CUSTODY FORM

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9484 Chesepeake Dr., Suite 805, San Diego, CA 92123 (818) 505-8596 FAX (818) 505-9689

Thiokol LWR E11712

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## **CHAIN OF CUSTODY FORM**

Client Name/Address:				PO Number:					Ana	lysis Red	uired					
Client Name/Address:  TPC  TPC  ZITECHNOLOGY I  TVING CA C  Project Manager:  Project Manager:  Chard Sco-  Sampler:	rive		98	-346 XKee	1										太	
Irvine, CAC	1261	8	LC	xilee	ed										M+X=	extrac
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Sample Description	Sample Matrix	Container Type	# of Cont.	Sampling Date/Time	Preservatives										Specia	l Instructions
PS-29   IB0355	5*	Glass	1	2/24/9	7										4/T UF	3/24
PS-30/ IB0355Z															<u> </u>	
PS-31/IB03553												<u> </u>				
PS-32/IB03554																
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25-34/IB03743	4	4		2/18/9	<b>*</b>										X H/T	3/18qu
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due within 30 days from the date of invoice. Sample(s) will be disposed of after 30 days.

# APPENDIX A.6 AIR FORCE RESEARCH LABORATORY

041499a Sequence Perchlorate eg401.0ml35mmoh-tds-ap Method 1000 uL Conc Area RT (ppb) 5.0 10688 17.25 Std 17.32 9151 5.0 Std 17.32 5.0 9143 Std 17.38 9556 Std 5.0 9935 17.17 Std 5.0 9874 17.17 5.0 Std 9835 17.17 5.0 Std 5.0 9635 17.17 Std 10.0 16415 17.40 Std 17.28 10.0 16712 Std 17.17 Std 10.0 17609 17.32 25.0 47817 Std 17.38 25.0 46827 Std 17.25 50.0 100957 Std 17.20 Std 100.0 217589 17.43 100.0 208748 Std 200.0 444062 17.10 Std 17.03 Std 500.0 1046409 17.03 Std 500.0 1055535 16.97 Std 500.0 1055124

MDL Calculations	
concentration (ppb) Average Standard Deviation %CV Student t-test (df = 7)	5 9727 462 5% 2.998
MDL (ppb) LRL (ppb) URL (ppb)	0.71 2.1 500

Fertilizer Extracts			Y=	2110.3		· · · · · · · · · · · · · · · · · · ·			
					Adjusted			Adjusted	Adjusted
			Diluted	F	for Dilution	25 ppb	1	For Dilution	or Dilution
Samples		Area	Conc,	Dilution		Spike	Duplicate		URL/D
ug/L	RT	Count	(ppb)	Factor	(ppm)	Recovery	% Recovery	(ppm)	(ppm)
LCS1	13.3	208776	98.93	1	0.099			0.002	0.5
LCS2	13.3	206792	97.99	1	0.098			0.002	0.5
ps1, 5000	17.1	201810	95.63	5000	478.2			11	2500
ps2, 2500	17.1	259763	123.09	2500	307.7			5	1250
ps3, 10000	17.2	162037	76.78	10000	767.8			21	5000
ps4, 5000	17.3	291546	138.15	5000	690.8			11	2500
ps5, 5000	17.4	291128	137.96	5000	689.8			11	2500
ps5, 5000, sp25ppb	17.4	339967	161.10	5000	712.9	939	%	11	2500
ps6, 5000	17.4	132036	62.57	5000	312.8			11	2500
ps6, 5000, dup	17.5	126539	59.96	5000	299.8		96%	11	2500
ps7, 5000	17.4	184404	87.38	5000	436.9			11	2500
ps8, 5000	17.4	33911	16.07	5000	80.3			11	2500
ps9, 5000	17.4	170872	80.97	5000	404.9			11	2500
ps10, Undiluted	13.2	Interference In	nterference	1	Interference			0	- 1
ps11, 5000	17.5	298312	141.36	5000	706.8			11	2500
ps12, 500	17.5	221964	105.18	500	52.6			ı	250
ps13, Undiluted	13.2	Interference I	nterference	1	Interference			0	1
ps14, 5000	17.5	291032	137.91	5000	689.6	)		11	2500
ps15, 5000	17.5	197232	93.46	5000	467.3	;		11	2500
ps15, 5000, sp25ppb	17.1	251038	118.96	5000	492.8	102	%	11	2500
ps16, 5000	17.6	261414	123.88	5000	619.4	<b>,</b>		11	2500
ps16, 5000, dup	17.1	263542	124.88	5000	624.4	,	101%	11	2500
ps17, Undiluted	17.6	Interference I	nterference	5000	Interference	:		11	2500
ps18, 500	17.5	381612	180.83	500	90.4	}		1	
ps19, 5000	17.4	86661	41.07	5000	205.3	3		11	2500
ps20, 5000	17.3	299576	141.96	5000	709.8	3		11	
ps21, 5000	17.1	100300	47.53	5000	237.6	<b>,</b>		11	2500
ps22, 1000	17.1	420941	199.47	1000		;		2	500
ps23, 1000	17.1	108413	51.37	1000	51.4	,		2	500
ps24, 5000	17.1	106784	50.60	5000	253.0	)		11	2500
ps25, 5000	17.1	157284	74.53	5000	372.7	7		11	2500
ps25, 5000, sp25ppb	17.0	210290	99.65	5000	397.8	3 100	%	11	2500
ps26, 5000	17.1	119622	<b>5</b> 6.68	5000	283.4	1		11	2500
ps26, 5000, dup	17.0	117618	55.74	5000	278.7	1	98%	11	2500
ps27, 5000	17.1	263617	124.92	5000	624.6	5		11	2500
ps28, 5000	17.1	132305	62.69	5000	313.5	5		11	2500
ps29, 5000	17.1	193132	91.52	5000	457.6	5		11	2500
ps30, 10000	17.1	288029	136.49	10000	1364.9	)		21	5000
ps31, 10000	17.0	247936	117.49	10000				21	5000
ps32, 5000	17.0	247408	117.24	5000	586.2	2	_	11	2500
ps33, 5000	17.0	303946	144.03	5000				11	2500
ps34, 25000	17.0	220549	104.51	25000	2612.8	3		53	12500
·									

Fertilizers													
Calculated based on adding	1 g of fertilizer	sample to 10	mL of water	•									
Extract													
Concentration		Extract	Amount	Amount	Conc.								
Samples	mg/L	Volume	ClO4-	Fertilizer	(g/kg)	Wt/Wt%							
ug/L	(ppm)	(L)	(mg)	(kg)	(ppt)								
ps1, 5000	478.2	0.01	4.782	0.001	4.78	0.48%							
ps2, 2500	307.7	0.01	3.077	0.001	3.08	0.31%							
ps3, 10000	767.8	0.01	7.678	0.001	7.68	0.77%							
ps4, 5000	690.8	0.01	6.908	0.001	6.91	0.69%							
ps5, 5000	689.8	0.01	6.898	0.001	6.90	0.69%							
ps6, 5000	312.8	0.01	3.128	0.001	3.13	0.31%							
ps7, 5000	436.9	0.01	4.369	0.001	4.37	0.44%							
ps8, 5000	80.3	0.01	0.803	0.001	0.80	0.08%							
ps9, 5000	404.9	0.01	4.049	0.001	4.05	0.40%							
ps10, Undiluted	ND		nterference		Interference								
ps11, 5000	706.8	0.01	7.068	0.001	7.07	0.71%							
ps12, 500	52.6	0.01	0.526	0.001	0.53	0.05%							
ps13, Undiluted	ND		nterference		Interference								
ps14, 5000	689.6	0.01	6.896	0.001	6.90								
ps15, 5000	467.3	0.01	4.673	0.001	4.67								
ps16, 5000	619.4	0.01	6.194	0.001	6.19								
ps17, Undiluted	ND		nterference		Interference								
ps18, 500	90.4	0.01	0.904	0.001	0.90								
ps19, 5000	205.3	0.01	2.053	0.001									
ps20, 5000	709.8	10.0	7.098	0.001									
ps21, 5000	237.6	0.01	2.376	0.001									
ps22, 1000	199.5	0.01	1.995	0.001									
ps23, 1000	51.4	0.01	0.514	0.001									
ps24, 5000	253.0	0.01	2.530	0.001									
ps25, 5000	372.7	0.01	3.727	0.001									
ps26, 5000	283.4	0.01	2.834	0.001									
ps27, 5000	624.6	10.0	6.246	0.001									
ps28, 5000	313.5	0.01	3.135	0.001									
ps29, 5000	457.6	0.01	4.576	0.001									
ps30, 10000	1364.9	0.01	13.649	0.001									
ps31, 10000	1174.9	0.01	11.749	0.001									
ps32, 5000	586.2	0.01	5.862	0.001									
ps33, 5000	720.1	0.01	7.201	0.001									
ps34, 25000	2612.8	0.01	26.128	0.001	26.13	2.61%							

## APPENDIX A.7

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY (TO BE PROVIDED)

APPENDIX A.8

CHEMICAL TESTING SERVICE (PERCHLORATE TITRATION RESULTS)

## **CTS**

## CHEMICAL TESTING SERVICE 23441 GOLDEN SPRINGS DIAMOND BAR, CA 91765 909-860-4100

Richard Scott TRC Environmental Solutions Inc 21 Technology Drive Irvine, CA 92618 April 18, 1999

## <u>LABORATORY REPORT</u> PERCHLORATE TITRATION RESULTS

Please find attached Tables 1 which summarizes the the results of the Triphenylstilbonium tritration for the perchlorate ion. Table 2 provides a summary of the ion-selective electrode analyses of the fertilizer samples conducted during the sample extraction process. A summary of the methods of analysis used in the studies is provided below. These methods are not state approved laboratory methods, and have been conducted to provide additional information on potential perchlorate interferences during Ion-Chromatograpy, and as a confirmatory analysis of Ion-chromatography results

### EXTRACTION METHODOLOGY

A 10 gram sample of the extract was diluted into 50 ml of distilled water and adsorbed onto 25 grams of Dowex 1 The column was eluted with 100 ml of resin (Boyd, 1968) potassium bicarbonate solution (0.01  $\underline{M}$ ) to remove nitrates, chloride, chlorite and chlorate ions. The wash solutions were monitored using a perchlorate selective electrode (Orion, Model 938101), to determine if a portion of the perchlorate eluted in the rinse. The extraction column was then rinsed with 75 mls of sodium fluoroborate solution (0.05 M) and the rinsed collected. The column was then rinsed with an additional 25 ml of sodium fluoroborate solution which was added to the previous eluate. The sodium fluoroborate solution was then analyzed using the perchlorate selective electrode. To determine the efficiency of the extraction process, various control samples and duplicate samples were also extracted and analyzed.

#### ANALYTICAL METHODOLOGY

The extracts produced above were analyzed using the Triphenylstilbonium titration method (Morris, 1965). A 10 ml aliquot of the extract was titrated with 0.01 M Triphenylstilbonium solution using bromocresol green as an indicator, in conjunction with the perchlorate selective electrode. The endpoint of the titration was clearly visible, with the bromocresol green color disappearing, producing a clear solution. At this endpoint, the perchlorate selective ion indicated no detectable perchlorate concentration.

#### REFERENCES

Boyd, G.E., Larson, Q.V., Journal American Chemical Society, (90), 5092, 1968.

Morris, M.D., Analytical Chemistry, (37), 977, 1965.

Note: The attached results should be considered as estimated quantities only, since this method has not been evaluated or approved by the California Department of Health Services.

TABLE 1
PERCHLORATE TITRATION RESULTS

SAMPLE ID.	TEST PARAMETER	RESULT	<u>UNITS</u>	MDL	DILUTION	ANALYST	TEST DATE
3512	CLO4-TITRATION	590	mg/l.	5	100	PCT	4/6/99
3513	CLO4-TITRATION	295	mg/l.	5	100	PCT	4/6/99
3514	CLO4-TITRATION	<50	mg/l.	5	100	PCT	4/6/99
3515	CLO4-TITRATION	680	mg/l.	5	100	PCT	4/6/99
3516	CLO4-TITRATION	715	mg/l.	5	100	PCT	4/6/99
3517	CLO4-TITRATION	340	mg/l.	5	100	PCT	4/6/99
3518	CLO4-TITRATION	544	mg/l.	5	100	PCT	4/6/99
3519	CLO4-TITRATION	125	mg/l.	5	100	PCT	4/6/99
3520	CLO4-TITRATION	520	mg/l.	5	100	PCT	4/6/99
3521	CLO4-TITRATION	<50	mg/l.	5	100	PCT	4/6/99
3522	CLO4-TITRATION	1060	mg/l.	5	100	PCT	4/6/99
3523	CLO4-TITRATION	75	mg/l.	5	100	PCT	4/7/99
3524	CLO4-TITRATION	1080	mg/l.	5	100	PCT	4/7/99
3525	CLO4-TITRATION	1035	mg/l.	5	100	PCT	4/7/99
3526	CLO4-TITRATION	614	mg/i.	5	100	PCT	4/7/99
3527	CLO4-TITRATION	698	mg/l.	5	100	PCT	4/7/99
3528	CLO4-TITRATION	<50	mg/l.	5	100	PCT	4/7/99
3529	CLO4-TITRATION	74	mg/l.	5	100	PCT	4/7/99
3530	CLO4-TITRATION	310	mg/l.	5	100	PCT	4/7/99
3531	CLO4-TITRATION	625	mg/l.	5	100	PCT	4/7/99
3532	CLO4-TITRATION	340	mg/l.	5	100	PCT	4/7/99
3533	CLO4-TITRATION	170	mg/l.	5	100	PCT	4/7/99
3534	CLO4-TITRATION	55	mg/l.	5	100	PCT	4/8/99
3535	CLO4-TITRATION	285	mg/l.	5	100	PCT	4/8/99
3536	CLO4-TITRATION	525	mg/l.	5	100		4/8/99
3537	CLO4-TITRATION	355	mg/l.	5	100	PCT	4/8/99
3538	CLO4-TITRATION	630	mg/l.	5	100	PCT	4/8/99
3539	CLO4-TITRATION	280	mg/l.	5	100		4/8/99
3551	CLO4-TITRATION	580		5	100		4/8/99
3552	CLO4-TITRATION	1560	mg/l.	5	100		4/8/99
3553		1680	<del>                                     </del>	5	100		4/8/99
3554	CLO4-TITRATION	820		5			4/8/99
3555		825		5	100		4/8/99
3793		3280		5			4/9/99
TRC-01	CLO4-TITRATION	970	mg/i.	5	100	PCT	4/9/99
TRC-02	CLO4-TITRATION	<50	mg/l.	5			4/9/99
TRC-03	CLO4-TITRATION	<50	mg/l.	5			4/9/99
TRC-04	CLO4-TITRATION	<50	mg/l.	5			4/9/99
TRC-05	CLO4-TITRATION	<50	mg/l.	5			4/9/99
BLANK	CLO4-TITRATION	<50	mg/l.	5			4/9/99
LCS-01	CLO4-TITRATION	90	<del></del>	5			4/9/99
LCS-02	CLO4-TITRATION	105	<del></del>	5			4/9/99
TRC-06	CLO4-TITRATION	475		5			4/9/99
TRC-07	CLO4-TITRATION	820		5			4/9/99
TRC-08	CLO4-TITRATION	630	<del></del>	5			4/9/99
TRC-09	CLO4-TITRATION	<50	mg/l.	5			4/9/99
TRC-10	CLO4-TITRATION	155	<del></del>	5			4/9/99
TRC-11	CLO4-TITRATION	320	) mg/l.	5	100	PCT	4/9/99

TABLE 2
PERCHLORATE ION-SELECTIVE ELECTRODE RESULTS

SAMPLE ID.	TEST PARAMETER	RESULT	<u>UNITS</u>	<u>MDL</u>	DILUTION	ANALYST	TEST DATE
3512	CLO4-SEL-ION	450	mg/l.	100	100	PCT	4/6/99
3513	CLO4-SEL-ION		mg/l.	100	100	PCT	4/6/99
3514	CLO4-SEL-ION	<100	mg/l.	100	100	PCT	4/6/99
3515	CLO4-SEL-ION	820	mg/l.	100	100	PCT	4/6/99
3516	CLO4-SEL-ION	650	mg/l.	100	100	PCT	4/6/99
3517	CLO4-SEL-ION	280	mg/l.	100	100	PCT	4/6/99
3518	CLO4-SEL-ION	620	mg/l.	100	100	PCT	4/6/99
3519	CLO4-SEL-ION	175	mg/l.	100	100	PCT	4/6/99
3520	CLO4-SEL-ION	440	mg/l.	100		PCT	4/6/99
3521	CLO4-SEL-ION	<100	mg/l.	100		PCT	4/6/99
3522	CLO4-SEL-ION	880	mg/l.	100		PCT	4/6/99
3523	CLO4-SEL-ION	<100	mg/l.	100		PCT	4/7/99
3524	CLO4-SEL-ION		mg/l.	100		PCT	4/7/99
3525	CLO4-SEL-ION	1120	mg/l.	100		PCT	4/7/99
3526	CLO4-SEL-ION	675	mg/l.	100	100	PCT	4/7/99
3527	CLO4-SEL-ION	640	mg/l.	100	100	PCT	4/7/99
3528	CLO4-SEL-ION	<100	mg/l.	100	100	PCT	4/7/99
3529	CLO4-SEL-ION	110	mg/l.	100	100	PCT	4/7/99
3530	CLO4-SEL-ION	280	mg/l.	100	100	PCT	4/7/99
3531	CLO4-SEL-ION	670	mg/l.	100	100	PCT	4/7/99
3532	CLO4-SEL-ION		mg/l.	100		PCT	4/7/99
3533	CLO4-SEL-ION		mg/l.	100		PCT	4/7/99
3534	CLO4-SEL-ION	<100	mg/l.	100		PCT	4/8/99
3535	CLO4-SEL-ION	345	mg/l.	100		PCT	4/8/99
3536	CLO4-SEL-ION	440	mg/l.	100	100	PCT	4/8/99
3537	CLO4-SEL-ION	350	mg/l.	100	100	PCT	4/8/99
3538	CLO4-SEL-ION	660	mg/l.	100		PCT	4/8/99
3539	CLO4-SEL-ION	330	mg/l.	100		PCT	4/8/99
3551	CLO4-SEL-ION		mg/l.	100		PCT	4/8/99
3552	CLO4-SEL-ION		mg/l.	100		PCT	4/8/99
3553	CLO4-SEL-ION	1755	mg/l.	100	100	PCT	4/8/99
3554	CLO4-SEL-ION	910	mg/l.	100	100	PCT	4/8/99
3555	CLO4-SEL-ION	775	mg/l.	100	100	PCT	4/8/99
3793	CLO4-SEL-ION		mg/l.	100		PCT	4/9/99
BLANK	CLO4-SEL-ION	<del></del>	mg/l.	100			4/9/99
LCS-01	CLO4-SEL-ION		mg/l.	100			4/8/99
LCS-02	CLO4-SEL-ION		mg/l.	100		1	4/9/99
TRC-01	CLO4-SEL-ION		mg/l.	100			4/9/99
TRC-02	CLO4-SEL-ION		mg/l.	100			4/9/99
TRC-03	CLO4-SEL-ION		mg/l.	100			4/9/99
TRC-04	CLO4-SEL-ION		mg/l.	100			4/9/99
TRC-05	CLO4-SEL-ION		mg/l.	100			4/9/99
TRC-06	CLO4-SEL-ION		mg/l.	100			4/9/99
TRC-07	CLO4-SEL-ION		mg/l.	100			4/9/99
TRC-08	CLO4-SEL-ION		mg/l.	100			4/9/99
TRC-09	CLO4-SEL-ION		mg/l.	100			4/9/99
TRC-10	CLO4-SEL-ION		mg/l.	10			4/9/99
TRC-11	CLO4-SEL-ION	415	mg/l.	100	100	PCT	4/9/99

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The material(s) listed are received for analysis and/or treatability evaluation and remain the property of the client and not TRC. At the conclusion of the test work, all remaining material(s) will be returned to the client for eventual disposal at a licensed facility.  TRC  2815 Mitchell Drive, Suite 103  Walnut Creek, California 9459  (925) 935-3294	

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# ATTACHMENT 1

DETERMINATION OF PERCHLORATE BY ION-CHROMATOGRAPHY

METHOD STATUS:

APPROVED FOR ROUTINE USE

METHOD REVISION NO.: 0

SRLB Northern Section Chief

Environmental Biochemist

Branch Chief

## DETERMINATION OF PERCHLORATE BY ION CHROMATOGRAPHY

## Scope and Application

- This method covers the determination of the perchlorate anion by ion 1.1 chromatography.
- The applicable matrices are shown below.
  - Drinking water, groundwater and reagent waters: This method has been found to perform adequately on water samples with conductivities up to 1000 µmhos/cm. Water samples with conductivities >1000 µmhos/cm have not been tested.
  - Although not specifically tested, this method is potentially applicable to surface water, mixed domestic water, and industrial wastewaters. See also Sect. 1.7.
- The Method Detection Limit (MDL defined in Sect. 3.10) and Reporting Limit (RL 1.3 defined in Sect. 3.12) for perchlorate in reagent water is 0.7 µg/L (pooled data) and 4.0 µg/L, respectively. See Table 1. The MDL and RL for a specific matrix may differ from that listed, depending upon the nature of the sample.
- 1.4 The linear calibration range for perchlorate is approximately 2.5 to 500 µg/L. Sample concentrations higher than the upper calibration limit should be diluted with reagent water to a concentration within the calibration range and reanalyzed.
- 1.5 Figure 1 shows the chromatograms for 4 ug/L of perchlorate added to reagent water and a groundwater sample.
- 1.6 This method is recommended for use by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.
- 1.7 When this method is used to analyze unfamiliar samples, perchlorate identification should be supported by the use of a fortified sample matrix. The fortification procedure is described in Sect. 11.5.
- 1.8 Users of the method data should state the data-quality objectives prior to analysis. Users of the method must demonstrate the ability to generate acceptable results with this method, using the procedures described in Sect. 9.

diff retain

June 3, 1997 CLO4METH SRL 1.9 <u>DISCLAIMER</u> Mention of trade names or commercial products does not constitute endorsement or recommendation for use. Equivalent product substitutions may be made by laboratories using this method as a reference.

# 2. Summary of Method

- 2.1 A fixed volume of sample is injected into an ion chromatographic system, where the perchlorate anion is separated from other interfering anions and quantified.
- 2.2 To detect perchlorate in the low ppb (µg/L) range without sample preconcentration, a high volume sample loop is used.
- 2.3 To minimize hydrophobic interaction of the perchlorate anion with the anion exchange support resin, p-cyanophenol is added to the eluent to deactivate the active sites on the resin. Without column deactivation, the perchlorate peak elutes with a longer retention time, is broader (isocratic elution), and tails severely, thus resulting in poor peak detection as the perchlorate sample concentration decreases.

# 3. Definitions

- 3.1 CALIBRATION BLANK (CB) A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.
- 3.2 CALIBRATION STANDARD (CAL) A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 3.3 FIELD DUPLICATES (FD) Two separate samples collected at the same time and location under identical circumstances and treated exactly the same throughout field and taboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
- 3.4 INSTRUMENT PERFORMANCE CHECK SOLUTION (IPC) A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.
- 3.5 LABORATORY FORTIFIED BLANK (LFB) An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the method is in control, and whether the laboratory is capable of making accurate and precise measurements.
- 3.6 LABORATORY FORTIFIED SAMPLE MATRIX (LFM) An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

- 3.7 LABORATORY REAGENT BLANK (LRB) An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
- 3.8 LINEAR CALIBRATION RANGE (LCR) The concentration range over which the instrument response is linear.
- 3.9 MATERIAL SAFETY DATA SHEET (MSDS) Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.
- 3.10 METHOD DETECTION LIMIT (MDL) The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. (See Ref. 16.3.)
- 3.11 QUALITY CONTROL SAMPLE (QCS) A solution of method analyte(s) of known concentration(s) that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.
- 3.12 REPORTING LIMIT (RL) The reporting limit used in this method is defined as the minimum quantifiable concentration level at which a sample concentration result may be reported and is equal to five times the MDL.

## 4. Interferences

- 4.1 Interferences can be caused by substances with retention times that are similar to and overlap the anion of interest. High concentrations of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.
- 4.2 The large water dip or negative peak is due to the large surquot of sample injected onto the column. However, the perchlorate anion is retained for a sufficient length of time in the column and eliutes free of interference from the water dip.
- 4.3 Due to the strength of the eluent, the majority of the anions in a water sample will elute soon after the water dip. Because of the large sample volume injected, the detector response from these anions may be very high, depending on the amount of dissolved solids present in the sample. With the longer retention time, the perchlorate anion elutes on the tail end of these early eluting anions and therefore, the detection and quantification of perchlorate is largely unaffected. See Figure 1.
- .4.4 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

4.5 Samples that contain particles larger than 0.45 microns and reagents solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

#### 5. Safety

- 5.1 The toxicity or carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.
- 5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.
- 5.3 The following chemicals have the potential to be highly toxic or hazardous. The MSDS for each chemical should be consulted.
  - 5.3.1 Sodium hydroxide (Sect. 7.2).
  - 5.3.2 Sulfuric acid (Sect. 7.3).
  - 5.3.3 Potassium perchlorate (Sect. 7.4).

# 6. Equipment and Supplies

- 6.1 Balance Analytical, capable of accurately weighing to the nearest 0.1 mg.
- 6.2 Ion chromatograph Analytical system complete with ion chromatograph and all required accessories including syringes, analytical columns, compressed gasses and detectors.
- 6.3 Sample loop: approximately 740 μL (12<sup>r</sup> x 0.02<sup>n</sup> l.D. tubing).
- 6.4 Anion guard column: Dionex IonPac AG5 (P/N 35396), or equivalent.
- 6.5 Anion separator column: Dionex IonPac AS5 (P/N 35395), or equivalent. This column produces the separation shown in Figure 1.
- 6.6 Anion suppressor device: Dionex AMMS-II (P/N 43074) suppressor system, or equivalent.
- 6.7 Conductivity detector -- Dionex CDM-II, or equivalent.
- 6.8 Chromatography data system: The data presented in this method were generated using the Dionex ACI-I computer interface and the Dionex AI-450 Data
   Chromatography Software. An equivalent data collection and chromatography processing system may also be used.
- 6.9 Sample bottles: polyethylene, 125 mL, or larger.

### 7. Readents and Standards

- 7.1 Reagent water. Distilled or deionized water, free of the anion of interest. The reagent water should contain particles no larger than 0.20 µm.
- 7.2 Eluent solution: 50% (w/w) Sodium hydroxide (CASRN 1310-73-2) 120 mM, p-cyanophenol (CASRN 767-00-0) 2.0 mM. Dissolve 19.20 g of 50% (w/w) sodium hydroxide (NaOH) and 0.4765 g of p-cyanophenol (NCC<sub>8</sub>H<sub>4</sub>OH, 95%, Aldrich P/N C9,400-9, or equivalent) in degassed reagent water and dilute to 2 L. The 50% (w/w) NaOH should be fresh with minimal contamination from dissolved CO<sub>2</sub> (carbonate formation).
- 7.3 Regenerant solution (micro-membrane suppressor): Sulfuric acid (CASRN 7664-93-9) 0.035N. Dilute 3.9 mL reagent grade conc. sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) to 4 L with reagent water.
- 7.4 Stock standard perchlorate solutions, 1000 mg/L (1 mg/mL): The stock standard solution is prepared from ACS reagent grade material. Dissolve 1.3931 g potassium perchlorate (KClO<sub>4</sub>, CASRN 7778-74-7) in reagent water and dilute to 1 L.
  - 7.4.1 Prepare a 1000 mg/L perchlorate (KClO<sub>2</sub>) stock solution for use in preparing the instrument calibration solutions and IPC solutions.
  - 7.4.2 Prepare a 1000 mg/L perchlorate stock solution using a material source different from that of the calibration stock for use in preparing the QCS, LFB and LFM. The QCS is used to verify the accuracy of the instrument calibration.
  - 7.4.3 The analyst should be aware of the purity of the potessium perchlorate used to prepare the stock standard. A weight correction must be made when the solid material is less than 99% pure.
- 7.5 Intermediate stock standard perchlorate solutions. Prepare 10, 1.0 and 0.10 mg/L. standard solutions from the stock standard solutions.
  - NOTE: Stability of standards: The stock standard is stable for at least one month when stored at 4°C. The intermediate stock and dilute working standards should be prepared weekly.

# 8. Sample Collection, Preservation and Storage

- 8.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.
- 8.2 Sample preservation and holding time for perchlorate determined by this method are as follows:

Analyte Perchlorate Preservation
Store at 4°C

Holding Time 28 days \* Note: Based on the stability of chlorate. Under the same conditions, the stability of perchlorate is expected to be equivalent to, or more stable than, chlorate.

## 9. Quality Control

- 9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.
- 9.2 Initial Demonstration of Performance
  - 9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS) and laboratory performance (determination of MDL) prior to performing analyses by this method.
  - 9.2.2 Linear Calibration Range (LCR) The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ± 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.
  - 9.2.3 Quality Control Sample (QCS) When beginning the use of this method, on a quarterly basis or as required to meet-data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within ± 10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.
    - 9.2.4 Method Detection Limit (MDL) The MDL must be established for the analyte, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit (Ref. 16.3). To determine the MDL value, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$MDL = (t) \times (S)$$

where.

- t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates].
- S = standard deviation of the replicate analyses.

MDLs should be determined every year, when a new operator begins work or whenever there is a significant change in the background or instrument response.

- 9.3 Assessing Laboratory Performance
  - 9.3.1 Laboratory Reagent Blank (LRB) The laboratory must analyze at least one LRB with each batch of samples. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL indicate that laboratory or reagent contamination should be suspected and corrective action must be taken before continuing the analysis.
  - 9.3.2 Laboratory Fortified Blank (LFB) The laboratory must analyze at least one LFB with each batch of samples. Calculate accuracy as percent recovery (Sect. 9.4.1.2). If the recovery of perchlorate falls outside the required control limits of 90-110%, perchlorate is judged out of control, and the source of the problem should be identified and resolved before continuing with the analysis.
    - 9.3.2.1 The laboratory must use LFB analyses data to assess laboratory performance against the required control limits of 90-110%. When sufficient internal performance data become available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

UPPER CONTROL LIMIT = x + 3S LOWER CONTROL LIMIT = x - 3S

- 9.3.2.2 The optional control limits must be equal to or better than the required control limits of 90-110%. After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to establish an on-going precision statement for the level of concentrations included in the LFB. These data must be kept on file and be available for review.
- 9.3.2.3 Replicates of LFBs should be analyzed quarterly, or sooner, to determine the precision of the laboratory measurements. Add these results to the on-going control charts to document data quality.
- Instrument Performance Check Solution (IPC) For all determinations the laboratory must analyze the IPC (a midrange check standard) and a calibration blank immediately following daily calibration, after every tenth sample (or more frequently, if required) and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is within ± 10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within ± 10%. If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be

discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data.

- 9.4 Assessing Analyte Recovery and Data Quality
  - 9.4.1 Laboratory Fortified Sample Matrix (LFM) The laboratory must perform a matrix spike on a minimum of 10% of the routine samples. In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The spiked perchlorate concentration must be high enough to be detected above the original sample concentration and should not be less than five times the MDL. The added perchlorate concentration should be the same as that used in the LFB.
    - 9.4.1.1 In a blind matrix spike, if the concentration of fortification is less than 25% of the background concentration of the matrix the matrix recovery should not be calculated.
    - 9.4.1.2 Calculate the percent recovery for perchlorate, corrected for the concentration measured in the unfortified sample, and compare the value to the initial LFM recovery range of 75-125%. Percent recovery may be calculated using the following equation:

$$R = \frac{C_A - C}{A} \times 100\%$$

where,

R = percent.recovery.

 $C_{\lambda}$  = fortified sample concentration.

C = sample background concentration.

A = concentration equivalent of analyte added to sample.

- 9.4.1.3 When sufficient internal performance data becomes available (a minimum of 20 analyses) develop control limits from percent mean recovery (X) and the standard deviation (S) of the mean recovery, as in Sect. 9.3.2.1.
- 9.4.1.4 "If the recovery of the analyte tails outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control (Sect. 9.3), the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related.
- 9.4.2 Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.
- 9.4.3 In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the use of an anion concentrator column, different columns and/or eluents, to improve the separation, quantification, or lower the cost of measurements. Each time

such modifications to the method are made, the analyst is required to repeat the procedure in Sect. 9.

9.4.4 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists over the identification of a peak in the chromatogram, confirmatory techniques such as sample dilution and fortification, must be used. Whenever possible, the laboratory should perform analysis of quality control check samples and participate in relevant performance evaluation sample studies.

# 10. Calibration and Standardization

- 10.1 Establish ion chromatographic operating parameters equivalent to those indicated in Table 1.
- 10.2 Prepare calibration standards at a minimum of five concentration levels and a blank by adding accurately measured volumes of one or more intermediate stock standards (Sect. 7.5) to a volumetric flask and diluting to volume with reagent water. Perform a full instrument calibration on a monthly basis, or whenever a significant change in instrument response is observed or expected.
  - 10.2.1 During this procedure, the perchlorate retention time must be recorded.
  - 10.2.2 To confirm the linearity of the calibration curve, the predicted concentration for each calibration standard should be calculated by using the established linear regression curve and response from each standard concentration. If the predicted response for any standard varies from the expected response by more than ±10%, perform corrective action.
- 10.3 The calibration curve must be verified by analyzing the IPC solutions on each working day, or whenever the anion eluent is changed, and after every 20 samples. If the response or retention time for perchlorate varies from the expected values by more than ± 10%, the test must be repeated, using fresh IPC solutions. If the resulfs are still more than ± 10%, a new calibration curve must be prepared.
- 10.4 Nonlinear response can result when the analytical column capacity is exceeded (overloading). The response of the detector to the sample when diluted 1:1, and when not diluted, should be compared. If the calculated responses are the same, the sample need not be diluted.

#### 11. Procedure

- 11.1 Table 1 summarizes the recommended operating conditions for the ion chromatograph. Included in the table is the estimated retention time that can be achieved by this method.
- 11.2 Check the system calibration daily and, if required, recalibrate as described in Sect. 10.
- 11.3 Analyze the IPCs, QCS, LRB, samples, LFB, LFMs, and blanks.

- 11.4 The width of the retention time window used to make the perchlorate identification should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.
- 11.5 If a sample concentration exceeds the calibration range, the sample must be diluted with reagent water to fall within the working range.
- 11.6 If the resulting chromatogram fails to produce adequate resolution, or if identification of the specific anion is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

NOTE: Retention time is inversely proportional to concentration. In some cases this peak migration may produce poor resolution or identification.

## 12. <u>Data Analysis and Calculations</u>

- 12.1 Peak integration may be performed using either the peak height or the peak area method. However, the method of peak height is frequently preferable to the method of peak area, as the peak height determination is generally less affected by baseline placement as compared to the peak area determination.
- 12.2 Examine the chromatograms for perchlorate baselines set by the parameters used in the chromatography method. Correct any baseline improperly set by the method by modifying the integration parameters in the method. Save the corrected baseline to the raw data file.
- 12.3 As a check on system performance, the response for a low concentration standard (e.g. 4 µg/L perchlorate standard solution) should be monitored and recorded. If the daily detector response is more than three standard deviations lower than the recorded mean response, perform corrective action.
- 12.4 Prepare the calibration curve by plotting the instrument response against the standard concentration. Compute the sample concentration (corrected for any sample dilution) by comparing the sample response with the standard curve.
- 12.5 Report only those values that fall between the Rt. (Table 1) and the highest calibration standard. Samples exceeding the highest standard should be diluted and reanalyzed.
- 12.6 Report the perchlorate results in ug/L.

#### 13. Method Performance

- 13.1 Table 1 gives the single laboratory MDL under the conditions listed.
- 13.2 Tables 2 and 3 give the single laboratory accuracy and precision for perchlorate in reagent water and in groundwater for the listed conditions.
- 13.3 Table 4 gives the single laboratory precision for replicate analyses of perchlorate ingroundwater samples.

## 14. Pollution Prevention

- 14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.
- 14.2 Quantity of the chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.
- 14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

### 15. Waste Management

15.1 The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further informalion on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Sect. 14.3.

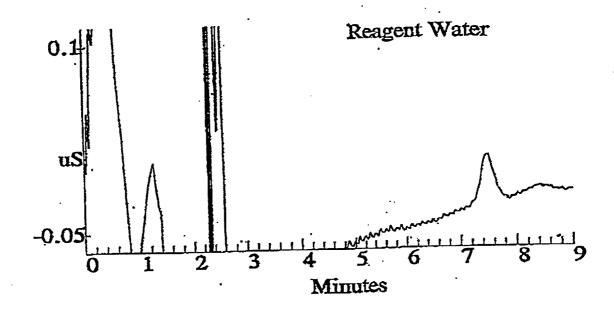
#### 16. References

- 16.1 Record 269, Dionex Chromatography Database 4.2.0, Dionex Corp., Sunnyvaia, CA, 94086.
- 16.2 Method 300.0, Revision 2.1, "Determination of Inorganic Anions by Ion Chromatography," August 1993, Environmental Monitoring Systems Laboratory, Office of Research and Development, USEPA, Cincinnati, OH, 45268.
- 16.3 Code of Federal Regulations 40, Ch. 1, Part 136, Appendix B.

#### 17. Acknowledgement

This method was developed and compiled by H.S. Okamoto, D.K. Rishi, S.K. Perera and F.J. Baumann of the California Department of Health Services, Division of Drinking Water and Environmental Management, Sanitation and Radiation Laboratories Branch. Technical advice was also generously provided by A. Fitchett, Dionex Corporation, Sunnyvale, California.

FIGURE 1. CHROMATOGRAMS OF 4 µg/L PERCHLORATE ADDED TO REAGENT WATER AND TO A GROUNDWATER SAMPLE



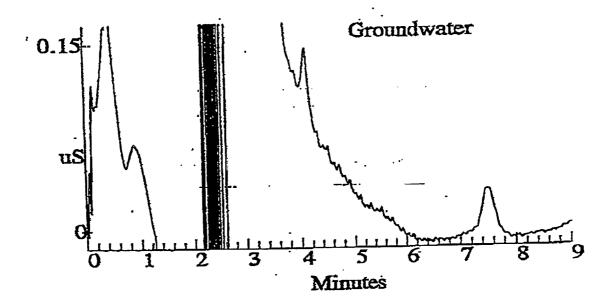


TABLE 1. CHROMATOGRAPHIC CONDITIONS AND DETECTION LIMIT IN REAGENT WATER

Perchlorate Spike Conc. (µg/L)	No. of Spiked Replicates	Mean Perchlorate Recovery (µg/L)	Standard Deviation (µg/L)	Calculated MDL (µg/L)
1.0	. 14	0.87	0.11	0.6
2.5 .	16	23	0.12	0.8
4.0 ·	16	3.9	0.11	0.7

Pooled MDL (df = 43)	0.7 µg/ւ_
RL (5 x MDL)	4 µg/L .
Retention Time	- 7.4 min.

Perchlorate peak height response for  $4.0 \mu g/L = 0.04 \mu S$ 

# Equipment and Standard Conditions Used to Produce Data in this Method:

Dionex 4500 Ion Chromatograph with Autosampier

Detector: Dionex CDM-2

Ion Suppressor: Dionex AMMS-II

Columns: Dionex IonPac AG5 Guard column (P/N 35396).

Dionex IonPac ASS Analytical (P/N 35395)

Column Temperature: Ambient

Injector Loop: 740 µL (approximate volume)

Eluent: 120 mM NaOH + 2.0 mM p-Cyanophenol

Eluent Flow rate: 1.0 mL/min.

Regenerant: 35 mN H,SO.

Regenerant Flow rate: 10 mL/min.

Conductivity Detector Background Reading: <12 µS

TABLE 2. SINGLE-OPERATOR ACCURACY AND PRECISION FOR PERCHLORATE STANDARD SOLUTIONS

		Known	Number	Mean Re	ecovery	60	500
Sample Type	Sample Matrix	Conc. (µg/L)	of Replicates	(µg/L)	(%)	SD (µg/L)	RSD (%)
		5.0	48	4.9	98	0.35	7.1
IPC Standard	RW.	100	47	100	100	4.2	4.2
		4.0	16	4.0	100	0.31	7.8
QCS	RW	100	4 .	100	100	2.8	2.8
LFB	RW	4.0	22	3.9	98	0.33	8.5

RW = reagent water

TABLE 3. SINGLE-OPERATOR ACCURACY AND PRECISION FOR PERCHLORATE MATRIX SPIKES

		Spike	Number		ecovery	Mean	SD of Mean
Sample Type	Sample Matrix	Conc. (µg/L)	of Spiked Pairs	(µg/L)	(%)	RPD (%)	RPD (%)
Matrix Spike/ Matrix Spike Duplicate	ĠŴ	. 4.0	20.	3.8	95	21	0.02

GW = groundwater

TABLE 4. SINGLE-OPERATOR PRECISION FOR PERCHLORATE SAMPLE REPLICATES

Sample Type	Sample Matrix	Number of Replicate Pairs**	Mean RPD (%)	SD of Mean RPD (%).
Sample/Sample Duplicate	GW	14	1.4	0.02

GW = groundwater

\*\*Note: Samples with perchlorate concentration > 4.0 µg/L